Sunken Treasure

Resurrecting a long-lost 19th-century fragrance.

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In February 2011, a strong winter storm exposed and removed a stable sediment layer deep within the intact bow section of the shipwreck Mary Celestia, a Civil War blockade runner sunk in 1864 in 20 meters of water off of the south shore of Bermuda. The removal of the sediment revealed a single bottle of corked wine and what appeared to be the corner of a wooden crate. The bottle was discovered as part of a routine survey of the wreck led by Philippe Max Rouja, Bermuda's custodian of historic wrecks, and local diver Stuart Joblin.

In June 2011, a team of local and international divers led by Rouja and colleagues James Delgado (director of the U.S. National Oceanographic & Atmospheric Administration's Maritime Heritage Program in the Office of National Maritime Sanctuaries) and Dominique Rissolo (archaeologist and executive director, Waitt Institute) conducted an archaeological excavation of the small section of the interior of the bow of the shipwreck. During the seven-day excavation the team uncovered and rescued a small cache of hidden artifacts, including a ship's taffrail log, a hair brush, shoes, and intact wine, cologne and perfume bottles. The cologne and the perfume bottles, like the wine, contained a visible air pocket above the liquid inside, suggesting that the contents had possibly remained uncontaminated over the intervening 150 years underwater.

Lost Fragrances

The cologne bottle was marked "Florida Water NY," while the perfume bottle was embossed with the lettering "Piesse & Lubin London."[a] Research by the team within the local diving fraternity that aided in the recovery revealed that specifi objects had been uncovered by several storms since Hurricane Emily in 1996. Several items matching those found during the current excavation had previously been recovered from deep within the bow section, including two intact and matching bottles from Piesse & Lubin. These were fortunately located in private collections and made available to the Government of Bermuda as needed.[b]

Initial research revealed that in the 1800s Piesse & Lubin was a prominent perfume house on Bond Street in London, England. G.W. Septimus Piesse was a chemist, perfumer and a prolific writer who wrote, among other scientific books, "The Art of Perfumery," the first modern book about perfumery, in 1857.[c]

Over seven days, an excavation team uncovered and rescued a small cache of hidden artifacts, including a ship's taffrail log, a hair brush, shoes, and intact wine, cologne and perfume bottles (pictured).

The archaeological team quickly made contact via email with perfume historian David Pybus (managing director, Scents of Time Ltd.), who helped pull together the history of the company and underlined that Piesse was a well-known and highly infl - ential figure in the development of perfumery in the late 19th century. Piesse was one of the first to openly describe and publish many of his highly influential techniques and formulas. It is not known if any examples of his work/perfume have survived to the present day, meaning that the specimens in the sealed and intact bottles from the Mary Celestia might represent the only known and testable samples of a Piesse perfume available to perfume historians and chemists.

An investigation was therefore launched to determine what, if anything, could be learned from the contents possibly preserved inside these 150-year-old bottles.

The Investigation Begins

Bermuda is fortunate to have a long and distinguished history in the development, manufacture and marketing of fragrances. Rouja reached out to the director of The Bermuda Perfumery, Isabelle Ramsay-Brackstone, to assist in planning the assessment and analysis of the perfume bottles and the liquid they contained. Brackstone instantly appraised the importance of the find and offered to pull together a team of international perfume experts to decide how to best proceed with the sampling and analysis.

In April 2013, two of the Piesse & Lubin perfume bottles recovered from the wreck of Mary Celestia, under special permission from the Government of Bermuda, were hand-carried

[a]The term "Florida water" was widely used in the 19th century to refer to American-made colognes that featured sweet orange, often in combination with spicy notes.
[b]Delgado Rouja et al., Mary Celestia report (In Press).
[c]The third edition of "The Art of Perfumery," written in 1862, mentioned the full gamut of odors derived from plants and applied to a range of scented products. The first edition was published in 1857; the fourth edition was published in 1880.

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by Brackstone from Bermuda to the laboratories of Drom Fragrances in New Jersey, where they were opened and their contents analyzed.

Description and Assessment of Bottles

In all, three bottles of fragrance identified as Piesse & Lubin were found in the bow section of the wreck of the Mary Celestia. The bottles were preserved 20 meters underwater, buried in dense sediment in the forepeak, the very tip of the ship. The conditions prior to disturbance by hurricane- and storm-driven ocean swells might be characterized as excellent. The bottles, which were 1 1/2 ounces (45 mL), were kept in a dark cool space under two atmospheres of pressure, with a constant temperature of about 19°C.

Sampling and Analysis

Drom Fragrances' analytical team was led by perfumer Jean-Claude Delville, technical perfumer Lionel Nesbitt and vice president Robert Stapf. Bottle 1 appeared to be "intact" in both appearance and content, with a small air pocket between the glass stopper and the liquid. Bottle 2 had a larger air pocket and therefore appeared to be missing about 15 mL of its contents. The liquid in Bottle 2 was slightly darker than Bottle 1, with traces of black sediment residue in the bottom of the bottle. The liquid in both bottles was distinctly light yellow in color and perfectly clear.

The glass stoppers appeared to be cemented and held firmly within the necks of the bottles. A thin dark line showed at the upper edge where the stopper merged with the neck and lip of the glass bottles. The neck was perfectly clean in Bottle 1, while there appeared to be some dried residue on the neck of Bottle 2. Both bottles were recovered from the shipwreck with the same level of liquid inside; it therefore appears that Bottle 2 likely developed a small leak post-recovery and some material evaporated, dried and calcified at the point of the leak. Fortunately, it appears that this "calcification" effectively resealed the bottle, but perhaps not before introducing some exchange of fresh or new air to the bottle.[d]

The thin mineral buildup on both necks may have been the result of submersion over the last 150 years and is most likely either a depositional or weathering product, reflecting the action of calcifying marine microorganisms or glass deterioration, respectively. Glass is not impervious to water. Over time and under pressure, it will allow some penetration of water, leading to glass deterioration. Glass objects of this period, especially if

[d]This egress of "fresh air" may have caused some minimal oxidization in specimen two and might account for the black residue inside, as well as the more pungent, or oxidized, smell or tone of the contents.

Wayne Lusardi, conservator at the U.S. National Oceanographic & Atmospheric Administration's Maritime Heritage division, inspects one of the uncovered fragrance bottles.

they are in salt water, can develop fine films of hydrated silica on their surface as the networks of glass are slowly broken down.[1] The development of such silica films may have in fact contributed to the long-term preservation of the contents as they potentially filled any gaps in the seal between the neck and the ground-glass stopper and certainly more securely held the stopper in place.

Both bottles were easily opened after these mineral deposits were carefully removed from around the glass stopper at their necks. Upon opening each bottle, a small amount (5 mL) of liquid was removed by pipette. This was transferred to a small glass vial and sealed with an inert cap by a lab technician. The glass bottles were then instantly resealed with their original glass stoppers.

The vials were labeled Specimen 1 and Specimen 2 and quickly made available for human analysis by olfactory experts, perfumers, and technological analysis by mass spectrometer and gas chromatograph. Olfactory analysis was carried out in the standard fashion: dipping a perfume blotter into each specimen vial until lightly coated with liquid, then waving it or passing it both at a distance and then progressively closer to the nose. The smells were instantly assessed, described and noted.

Simultaneously, 1 mL of liquid was taken via pipette from each of the specimen vials and transferred to an Agilent 5975C mass spectrometer via standard dipping stick and analyzed for chemical qualification via an Agilent 6890 dual column FID gas chromatograph.

Olfactory Results

Specimen 1 smelled principally of rotten orange, bergamot and grapefruit, with some background notes or impressions of rose and geranium. Specimen 2 had a more pronounced smell than Specimen 1, giving similar rotten citrus impressions, but also containing a strong, lasting and eventually overwhelming note of hydrogen sulfide. Animalic notes of ambergris,

civet and castoreum were also perceived. Some floral notes of neroli, orange flowe , orris and geranium could be perceived, as well as woodsy notes of bois de rose, sandalwood, vetiver and benzoin. Ambery notes of opoponax, styrax, labdanum resinoid and incense were also recognized. Both specimens had top note impressions of orange oil and petitgrain. The middle notes were perceived to be of natural rose and geranium. An orange flower absolute with traces of civet or ambergris tincture were recognized in the dry down.

Chemical Analysis Results

A comparison of the results for both specimens from the mass spectrometer and gas chromatograph revealed that the chemistry of the fragrances inside were exactly the same.[e]

The ingredients found in the samples are listed in formula LFN572 (**T-1**).

The citrus portion of the fragrance may have broken down or been distilled in a manner that most facilities do not use in contemporary perfumery. There was no presence of myrcene or p-cymene. A large quantity of p-cymene would indicate citrus breakdown. Based upon the terpinenes found, as well as the amount of limonene, it is clear that a combination of lemon and mandarin was used. Limonene was added to balance the difference between the formula and the target. It is very possible that the original sample contained grades of citrus oils that no longer exist. If the sample tested had revealed the presence of linalyl acetate, the researchers could have assumed that bergamot oil may have been the source of citrus in the fragrance; however, that ingredient was not found. The linalool therefore may have originally been derived from bois de rose or, possibly, another natural ingredient derived from rose oil. There is also a substantial amount of phenyl ethyl alcohol, which may have been derived from rose oil. The ethyl ether ingredients are breakdown products of the associated esters (linalyl ethyl ether, terpinyl ethyl ether, geranyl ethyl ether and α-terpinyl ethyl ether).

The hydrocarbon ingredients ethyl myristate, ethyl palmitate, ethyl heptadecanoate, ethyl stearate, ethyl oleate, ethyl linoleate and ethyl linolenate are usually associated with resins and absolutes. It is very possible that they are derived from rose oil. It is also possible that they may have been derived from an osmanthus absolute. Several acids (myristic, palmitic and stearic) discovered in the sample are found in absolutes and resins as well. Ingredients from a sulfury or blackcurrant oil were not found, despite the specimens' strong odor when the bottles were opened. However, that does not rule out that these ingredients were used. It was not possible to determine which absolutes or resins were initially used in this fragrance as many of them contain similar ingredients.

Mystery Identity

At this time, the qualitative and chemical analysis of the fragrance found in the Mary Celestia did not allow the researchers to determine the identity of the fragrance with certainty. The earliest reference of the collection of fragrance of Piesse & Lubin dates back to 1873 (**T-2**).

[e]It is important to note that the results make it absolutely clear that there had been no egress of salt water in these bottles, which would have registered on the chromatograph or affected the pH. In short, the researchers did not chemically detect the presence of salt water in the samples.

T-1. Formula LFN572

Description	Quantity
Benzyl alcohol	3.50
Citronellol	30.00
Geraniol pure	55.00
Geranyl acetate 60	17.50
Geranyl ethyl ether	10.00
Lemon oil	55.00
d-Limonene	60.00
Linalool (synthetic)	142.00
Mandarin oil green extra	15.00
Nerol	25.00
Phenylethyl alcohol	90.00
Terpinen-4-ol	2.00
Terpineol	25.00
Total	**530.00**

T-2. The known fragrance collection of Piesse & Lubin

Fragrance	Year Launched
Ambergris	1873
Hungary Water	1873
Kiss Me Quick	1873
Opoponax	1875
The Flower of the Day	1875
White Rose	1875
Frangipani	1880
Kisses	1880
Myrtle	1880
Frolic	1894
Musk-Deer	1900
Ribon de Bruges	1900
Vashti	1900

Source: Perfume Intelligence: The Encyclopedia of Perfume—as listed in the London Post Office Directory of 1899; *www.perfumeintelligence.co.uk/library/perfume/p/houses/Piesse.htm*

The researchers' review of the limited literature available about Piesse leads them to believe that the fragrance contained in the bottles found was probably an ancestor or precursor to one or many of the fragrances listed in **T-2**. In the literature, it appears that *Bouquet Opoponax* was the most popular fragrance marketed by Piesse & Lubin at the time that Mary Celestia wrecked. According to Octavian Coifan, perfume historian (*1000fragrances.blogspot.com*), *Bouquet Opoponax* was launched around 1859 and was outstandingly popular. Piesse was a chemist with an excellent aptitude for marketing. He understood that describing fragrances by their olfactory "impressions," rather than by their actual ingredients, had much more appeal with his customers. According to Coifan, the ingredient description of *Bouquet Opoponax* (without percentages of each ingredient in the formula) was as follows:

> Opoponax resin, vanilla resin, tonka bean resin, lemon oil, bergamot oil, mandarin oil, patchouli oil, civet

absolute 10%, Egyptian jasmine absolute, rose de mai absolute, orris concrete 1%, olibanum resin 10%.

There is no doubt that the techniques and solvents used by Piesse to extract the oils, absolutes, concretes and resins, as well as the natural aging influences over the past 150 years, have altered the original chemical composition of the ingredients of the fragrance. Therefore, while the analysis of the samples performed by Drom may be at the leading edge of perfumery's technological ability, it is impossible to confidently determine the exact original chemical composition of all the ingredients that have come together to make up this fragrance. The results, however, leave the authors with no doubt that the chemistry of these perfume samples closely matches what one would expect to find based on the historic literature on Piesse's formulas and descriptions.

According to Jean Kerléo, perfumer and cofounder of the Osmothèque, the scent archive in Versailles, France, the historical value of the fragrance found in the recovered bottles of Piesse & Lubin will only become relevant if the original formulae are found, as well as the methods used to extract the essential oils used to create the fragrance. With continued research of the literature and history of Piesse & Lubin, the authors could potentially better understand the mysteries found in this ancient bottle.

As mentioned, Piesse was not just a prominent chemist and perfumer of his time. Today, he is known and remembered not for his fragrances, per se, but for his authoring "The Art of Perfumery." In the third edition of his book, published in 1862, Piesse suggests that "scents, like sounds, appear to influenc the olfactory nerve in definite degree" and that there might be "octaves" of odors. Piesse believed that scents could be put together harmoniously, like musical notes. This analogy to music is still used today to describe fragrances by the volatility of their components, such as top, middle and bottom notes.

Although the perfume house never received the prestigious "Royal Warrant seal" from the British government, it is clear that Piesse's fragrances were admired and coveted even in the American South at the height of the American Civil War.

Drom perfumers Jean-Claude Delville (left) and Lionel Nesbitt were involved in the recreation of the lost fragrance. Upon opening each discovered bottle, a small amount of liquid (5 mL) was removed by pipette.

Through this fortuitous discovery and our analysis, the authors hope to add another clear note to the opus that is Piesse's legacy.

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