



# Toxicological analysis of a "poison vial" found in the remains of an SS soldier (Maltot, Normandy, France)

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## Abstract

In Maltot (Normandy, France), one grave containing the remains of a German soldier, who died in 1944, was excavated amongst other graves and isolated elements. A dozen whole vials were unearthed, resulting in questions about their content. Various screenings were carried out on the contents of one single vial: HPLC–DAD and HR-LC–MS screening after 1/10 dilution in mobile phase, GC–MS and HS-GC–MS after 1/10 dilution in methanol, multi-element research by HR-ICP-MS after total mineralization, and cyanide analysis. Analyzed vial contained approximately 300 µL of a colorless, water-immiscible liquid with a characteristic solvent odor. HPLC–DAD, GC–MS, HR-LC–MS/MS, ICP-MS, and cyanide screenings were negative excluding the presence of cyanide, arsenic, barbiturates, amphetamines, or narcotics. HS-GC–MS analysis highlighted the presence of ethanol, chloroform, and diethyl ether at significant concentrations. Chloroform and diethyl ether were anesthetic products mainly reserved for urgent situations. We hypothesized that the soldier may have been a combat medic working on battlefields. as he was wounded, another possibility could be that he may have used the vials to relieve his pain; however, the immediate severity of the wounds drove us to assess the second hypothesis of delayed death as being less plausible. The high number of vials containing ethanol, chloroform, and diethyl ether, and the massive blood loss leading to quick death led us to support the combat medic or paramedic hypothesis.

**Keywords** Forensic · Anesthesia · Chloroform · WWII · GC–MS · Toxicology

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## Introduction and purpose

Contemporary archaeology, which includes in it the remains of recent world conflicts, has long been thought to be too recent and too abundantly archived (written sources, video and photographic media, memory of the living, etc.) to be considered useful for historical studies or interpretations.

In Maltot (near Caen, Normandy), three graves containing disturbed remains of British soldiers, one grave containing the remains of a German soldier, and, within some distance, an isolated foot in a (British) shoe as well as the remains of an isolated hand were excavated during archaeological examination of an older site (Iron Age). The identification of the original nationality was based on the associated military equipment (pieces of uniform and fighting accessories).

These remains were probably related to the events of July 10–11, 1944 (battle of Hill 112), occurring shortly after the Normandy landings.

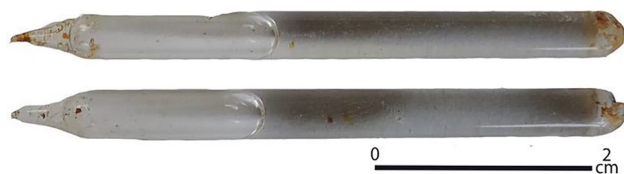
A dozen whole vials were unearthed at the thoracoabdominal level of the German soldier (Fig. 1).

We tried to determine the content of the vials through chemical analysis, hypothesizing they may have been filled with cyanide, as we know cyanide was the preferred poison for a wave of Nazi suicides following their defeat [1, 2].

## Material and method

### Chemicals and reagents

Prazepam was from Lipomed (Arlesheim, Switzerland). Formic acid, sulfuric acid, and acetonitrile were from VWR (Saint-Prix, France). Chloroform and diethyl ether were purchased from Carlo Erba (Val-de-Reuil, France). Sodium dihydrogenophosphate, ammonium formate, methanol, naphthalene 2–3 dicarboxaldehyde (NDA), taurine, ethanol, propan-2-ol, 1-propanol, and dimethylsulfoxide (DMSO) were supplied by Merck (Saint-Quentin-Fallavier, France). All solvents were gradient grade except acetonitrile, which was LC–MS (liquid chromatography–mass spectrometry) grade. Ultra-pure water (18 M $\Omega$ V) was obtained by ultrafiltration



**Fig. 1** Two out 15 vials found on the German soldier (Maltot, Normandie)

using a Milli-Q Integral device (Merck, Fontenay sous Bois, France).

### Analytical procedure

Various screenings were carried out on the contents of the vial: HPLC–DAD (high-performance liquid chromatography with diode array detector) and HR (high resolution) LC–MS screening after 1/10 dilution in mobile phase, GC–MS (gas chromatography–mass spectrometry) and HS–GC–MS (headspace GC–MS) after 1/10 dilution in methanol, multi-element research by HR–ICP–MS (high resolution–inductively coupled plasma–mass spectrometry) after total mineralization, and cyanide analysis.

### HPLC–DAD screening

HPLC–DAD screening was carried out on an Alliance 2695 chromatograph with a 2996 Diode Array detector (Waters, Guyancourt, France). Separation was performed on a Symmetry C8 column (250 × 4.6 × 5  $\mu$ m, 5  $\mu$ m) (Waters, Guyancourt, France) using a gradient of phosphate buffer 20 mM pH 3.8 (phase A) and acetonitrile (phase B). Briefly, the gradient started at 15% of B, then increased to 35% in 9 min followed by an increase to 80% at 28 min. Composition was held during 3 min before returning to initial conditions.

UV spectra were acquired between 190 and 450 nm and were identified using Toxicol™ library (Waters) and a home-made library using Empower 2 Software (Waters, Guyancourt, France).

### GC–MS screening

GC–MS analysis was performed on a Focus GC system equipped with a Triplus Duo autosampler. The detector was a DSQ II simple quadrupole (Thermo Fisher Scientific, Les Ulis, France). Separation was achieved using an UptiBond 5 (5% phenyl–95% dimethylpolysiloxane) column (Interchim, Montluçon, France) (30 m × 0.25 mm, dF = 0.25  $\mu$ m) with helium as carrier gas at a constant flow rate of 1.2 mL/min. Injection mode was splitless and injector temperature was maintained at 250 °C. The GC conditions were as follows: column temperature began at 45 °C, was held for 1 min, then increased to 115 °C at a rate of 30 °C/min, then held at 115 °C for 2 min before increasing to 300 °C at a rate of 3 °C/min and was held 14 min. Data acquisition was performed using the Xcalibur v2.1 software (Thermo Fisher Scientific). The MS was operated in full scan with an *m/z* range from 30 to 560. Data were processed using three different MS library: N.I.S.T. library, Maurer, Pfleger, and Weber v3, and SWGDrug v3.6.

### Volatile organic compound screening using HS-GC–MS

Research of volatile products was performed using previously described GC–MS, equipped with a Triplus AS Head space injector (Thermo Fisher Scientific). Separation was achieved on a Rtx-VMS column (30 m × 0.25 mm, dF = 1.4 μm) (Restek, Lisses, France) with helium as carrier gas at a constant flow rate of 1.2 mL/min. 5 μL of the sample was put in a sealed vial then heated to 80 °C during 20 min with a 10-s agitation step, then 0.2 mL of the gas phase was injected in the GC/MS. Injector temperature was maintained at 65 °C and split injection mode was employed with a split flow of 10. The GC conditions were as follows: column temperature began at 40 °C, was held for 2 min, and then increased to 150 °C at a rate of 10 °C/min then held for 1 min. 5 μL of the sample was put in a sealed vial then heated to 80 °C during 20 min with a 10-s agitation step. 0.2 mL of the gas phase was injected in the GC/MS. Data acquisition was performed using the Xcalibur v2.1 software (Thermo Fisher Scientific). The MS was operated in full scan with an *m/z* range from 10 to 250. Data was analyzed against three different MS libraries: N.I.S.T. library, Maurer, Pflieger, and Weber v3, and SWGDrug v3.6.

### LC-HRMS screening

LC-HRMS (liquid chromatography-high resolution mass spectrometry) was performed using a previously described method [3].

### Multi-element screening using ICP-MS

Multi-element screening was realized according to our previously published methodology [4].

### Cyanide quantification

The cyanide ion eventually contained in sample is derivatized in an alkaline medium by NDA (naphthalene 2–3 dicarboxaldehyde) and taurine in the presence of concentrated sulfuric acid. The derivative obtained is diluted in 100 μL of a 20 mM formate/acetonitrile buffer mixture (50/50, v/v). 10 μL are injected into the chromatographic system which consists in a G1312A binary pump (Agilent) hyphenated with a TSQ Quantum Access MAX Mass spectrometer (Thermo Fisher Scientific). A HyPURITY C18 column was used for chromatographic separation (150 × 2.1 mm 5 μm) (Thermo Fisher Scientific). The mobile phase consisted of a gradient of formate buffer and acetonitrile.

### Volatile organic compounds quantification

Quantification of volatile organic compounds was performed using the GC–MS method devoted to volatile organic compound screening ("[Volatile organic compound screening using HS-GC–MS](#)"). Injector temperature was maintained at 65 °C and split injection mode was employed with a split flow of 80 instead of 10. The MS was operated in SIM mode. Propan-1-ol was used as internal standard (E.I.).

### Working solutions, calibration standard, and quality controls

Dilution of chloroform, diethyl ether, ethanol, and isopropanol was performed in DMSO in order to get a 1 g/L stock solution. Five calibrator levels were prepared to produce calibration curve by spiking with appropriate volumes of the previously mentioned working solutions in 100 μL of DMSO. Calibration levels were 50, 100, 200, 500, and 1000 mg/L. QC samples were also prepared in blank whole blood at concentrations of 75, 300, and 750 ng/mL, and a zero calibrator was prepared. Because of the high concentration of the analyzed unknown liquid, sample had to be diluted 1:100 and 1:1000. Dilution was performed in DMSO.

## Results

### Archaeological context

The body of the German soldier was found in a grave located 80 cm deep from the current surface, measuring 180 × 75 cm, oriented East–West (Fig. 2). The skeleton was on its stomach, upper left limb in extension placed above the head. Biologically, according to the synostosis processes of the cranial sutures, the dental eruption pattern, and the intermaxillary suture, the individual would have been between 17 and 20 years old. The estimated height was around 1.72 m [5]. Pelvic analysis identified a male subject.

Numerous clothing artifacts were in contact with the deceased: aluminum jacket buttons, shirt buttons, leather trouser suspenders, fragment of lamp glass, remains of a gas mask attachment, and cigarette holder containing French coins. At the level of the trouser pockets, personal items were identified: pious medal and girdle containing a small Blessed Virgin of Lourdes, a comb, a button, a condom, a metal box, two warheads of 9 mm German parabeulum bullets, and a British casing ammunition. Finally, at the level of the abdomen, in what could possibly be a clothing pocket, the remains of a leather and metal box were unearthed, containing about ten whole vials, while 2 others lay loose at the level of the thorax.



**Fig. 2** Body of the German soldier, found in prone position in a pit located 80 cm deep from the current surface and measuring 180×75 cm, oriented East–West (Maltot, Normandy); photo: E. Tribouillard (INRAP)

### Volatile organic compounds quantification

The quantification method was linear from 50 to 1000 mg/L with a limit of detection of 10 mg/L. All the coefficients of variation of the QC samples prepared at 3 concentrations (75, 300, and 750 ng/mL) evaluated 6 times a day for 3 days were all below 15%, with an accuracy greater than 90%.

### Unknown sample

Analyzed vial contained approximately 300  $\mu$ L of a colorless, water-immiscible liquid with a characteristic solvent odor. HPLC–DAD, GC–MS, HR-LC–MS/MS, ICP–MS, and cyanide screenings were negative excluding the presence of cyanide and inorganic elements such as arsenic and drugs such as barbiturates, amphetamines, and narcotics. HS-GC–MS analysis highlighted the presence of several volatile products (Fig. 3). Ethanol, chloroform, and diethyl ether have been identified at significant concentrations in the liquid, while trace levels of isopropanol, trichloroethylene, and tetrachloroethylene were also found. Results of the quantification of volatile compounds are provided in Table 1.

### Discussion

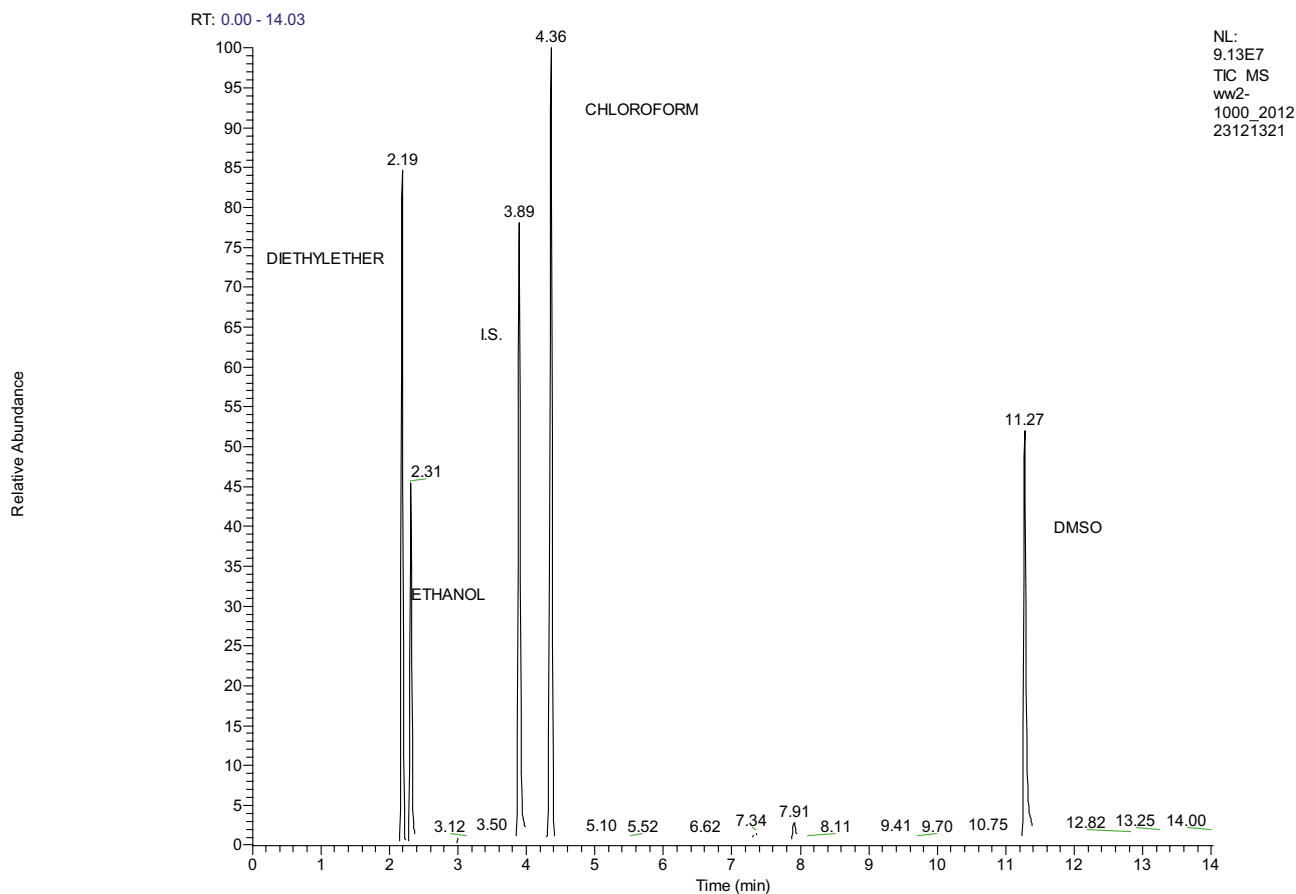
No poison was detected in this vial: no cyanide, arsenic, or other product that would cause short-term death. No barbiturates, amphetamines, and narcotics were identified either.

Benzodiazepines were also searched for but, as expected, were not found since the first molecule of the class was identified only in 1955 [6].

However, two anesthetic substances (chloroform and diethyl ether) associated with an alcoholic solvent (ethanol) were present.

Chloroform and diethyl ether are both colorless volatile fluids. Chloroform has been widely used since the American Civil War for its numbing properties, either by itself or in combination with ether. It is understood that the violence of the fighting indeed required rapid anesthesia on the battlefield: historians have estimated that almost 125,000 operations or procedures were performed under anesthesia during the Civil War, meaning that 25% of injured American soldiers received anesthesia [7]. Union armies data relying on 8900 major operations demonstrated that chloroform was the most widely used anesthetic [7]. The Confederate Cavalry specifically raided Union supply trains for the prized possessions of food and chloroform [8]. At the beginning of the twentieth century, chloroform was thought to exert effect eight to ten times faster, more complete and more intense than that of ether [9].

Chloroform was administered through inhalation. Chloroformic anesthesia began with a phase of “drunkenness” for the injured patient, if not a feeling of suffocation. The wounded sometimes even struggled to try to remove their masks. When this phase was over, breathing became calmer, more regular, then deep sleep occurred [10]. The use of chloroform continued until the 1940s. Unlike other anesthetics, it was easy to carry and frequently used in the battlefields of the Second World War (WWII).



**Fig. 3** HS-GC–MS chromatogram of the liquid (DMSO used for dilution)

As for chloroform, the main advantage of ether was also its form of administration: inhalation, which induced general anesthesia. The inhalation mode used either a drip impregnated compress or a mask that was applied to the nose and mouth of the injured person connected to a rubber balloon containing ether and air [9].

It is believed that up to the end of WWII, less than 10% of the general anesthetics administered in the USA were intravenous barbiturates [11]. The remaining 90% of anesthetics given were diethyl ether. Diethyl ether administration was a relatively safe and simple procedure, often delegated to

nurses or junior doctors with little or no specific training in anesthesia [11].

In the German medical practices during WWII, the use of chloroform and ether was mainly reserved for urgent situations and battlefields. Barbiturates were widely employed intravenously in operating theaters far from the front line. Ether was used for inhalation anesthesia except in cases presenting pulmonary infection, in which chloroform was used [12].

An interesting fact to underline is that in Germany, after 1905, a mixture known as “A.C.E,” consisting of 1 part alcohol, 2 parts chloroform, and 3 parts ether, became preferable to chloroform only [11]. It was believed that the myocardial depression produced by chloroform could be reduced by the stimulus of ether and alcohol. While chloroform was seldom used in England and the USA after the turn of the century, it remained popular in Germany until 1910, and the A.C.E. mixture remained in use for even longer [13].

The composition of the vial we analyzed matches with the general description of an A.C.E mixture, although quite different in terms of the exact repartition of fluids: 4.1 parts alcohol, 4.5 parts chloroform, and 1 part ether.

**Table 1** Concentration of the identified molecules in the liquid

	Concentration
Ethanol	360 g/L
Chloroform	392 g/L
Diethyl ether	87 g/L
Isopropanol	<5 g/L
Trichloroethylene	<5 g/L
Tetrachloroethylene	<5 g/L

We tried to determine whether inhaling more than one vial could have fatally intoxicated the soldier: the inhalation of high concentrations of chloroform mainly induces central nervous system depression; at high exposure levels (40,000 ppm), death can occur within minutes; concentrations between 1,500 and 30,000 ppm induce anesthesia, and lower levels (< 1,500 ppm) cause fatigue, dizziness, and headaches [14].

Literature data are nonetheless incomplete. Fragments of information on the exposure levels of chloroform leading to death in humans were obtained from clinical reports of patients exposed to chloroform as a method of anesthesia. A 113-min exposure at a concentration of 8,000 ppm provoked arrhythmia, vomiting, and increased prothrombin time in one patient; a 0.5–2-h exposure at 22,500 ppm provoked changes in respiratory rate, cardiac arrhythmia and bradycardia, vomiting, and transient jaundice in another patient [13].

The major effects caused by diethyl ether are narcosis, anesthesia, and central nervous system depression [15]; no data could be found in the literature regarding the level and duration of inhalation exposure which could lead to death in humans. The lethal concentration was 42,000 ppm (127,4 mg/L) for a 3-h exposure in mice, and 32,000 ppm (approx. 97 mg/L) for a 4-h exposure in rats [15].

Case reports of suicide with ether or chloroform failed to be informative because the quantities of toxicants inhaled could not be precisely known (volatility of solvents), and deaths resulted primarily from deliberate anoxia [16]. Consequently, we could not discuss the potential toxicity of inhaling more than one vial.

The forensic examination mentions a severe upper arm injury with major deterioration of the surrounding structures, and so we can assume the resulting massive hemorrhage led the soldier to his death in the very minutes or hours following the shock. Thus, the vials found on his body would not be correlated to the humeral wound: the soldier may have been a nurse or a paramedic posted on battlefields to help doctors perform emergency surgeries. The number of anesthetic vials unearthed is an argument in favor of this hypothesis. However, no decisive proof was found: no helmet was excavated, as unearthing the usual combat medic's helmet featuring the iconic red cross would have been an unquestionable proof. Neither an armband with the red cross, intended to dissuade enemy shelling, was found (garments were all destroyed through decades). The job of combat medics or paramedics was not to conduct elaborate and extensive treatment of the wounded, but to stabilize them and prepare them for evacuation to field hospitals or medical centers to the rear. Medics from both Allied and Axis nations performed the same basic functions [17].

Since we cannot ascertain the death occurred in the moments following the shock, another possibility would be to consider the soldier did not die immediately and survived

for a few days. In this case, it is legitimate to think that the vials found on the soldier were given to him to relieve his pain and keep him in a stable state with a view to treating him later. However, we believe this hypothesis of delayed death to be less plausible, because of the blood loss which is likely to have been considerable, and the high number of vials found.

## Conclusion

From the combined chromatography and ICP coupled with high resolution mass spectrometry analysis, we could establish the vials found on a German soldier who died in July 1944 on the Normandy front contained neither cyanide — as first anticipated — nor inorganic elements such as arsenic, nor drugs such as barbiturates, amphetamines, or narcotics. The analysis identified ethanol, chloroform, and diethyl ether at significant concentrations in the liquid, while traces of isopropanol, trichloroethylene, and tetrachloroethylene were also found.

The forensic examination attributed the cause of death to a polytrauma of the upper right limb, likely to have provoked a massive hemorrhage. It is not clear whether the 12 vials that were found aimed at relieving the soldier of pain, or if the soldier himself was a field paramedic in charge of delivering first aid to the wounded. The number of vials and the massive blood loss probably followed by quick death are two elements which led us to support the paramedic hypothesis.

The absolute necessity of anesthetic products during bad situations and the potential risk of their misuse highlight the role of anesthetists as medical specialists. Anesthesia developed as a medical discipline in Great Britain during the nineteenth century because of the interest and support of physicians and the complexity of administering chloroform anesthesia [15]. Post-WWII physician-anesthetists showed they could provide complex anesthesia care, regional anesthesia, and tracheal intubation, gaining the support of surgical colleagues who facilitated their growth within the medical profession [18].

## Key points

1. The body of a German soldier was found in Maltot (Normandy, France) in a grave located 80 cm deep from the current surface, and carrying at the level of the abdomen about ten whole vials, while 2 others lay loose at the level of the thorax.
2. We used HPLC–DAD and HR-LC–MS screening, GC–MS and HS-GC–MS, multi-element research by HR-ICP-MS, and cyanide quantification to determine the content of the vials.

3. HS-GC–MS analysis highlighted the presence of ethanol, chloroform, and diethyl ether at significant concentrations.
4. Other screenings were negative, excluding the presence of cyanide, arsenic, barbiturates, amphetamines, or narcotics.
5. Because of the numerous vials the soldier carried, he may have been a nurse or a paramedic on battlefields to help doctors perform emergency surgeries.

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**Author's contributions** All authors contributed to the study conception and design. All authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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## Declarations

**Ethical approval** No particular ethical approval was required.

**Informed consent** Not applicable.

**Research involving human and animal participants** This is a forensic report supporting archeological searches, for which a permit was granted and which performed with respect to the human body.

**Conflict of interest** The authors have no relevant financial or non-financial interests to disclose.

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