**Effects of Salinity and Temperature on seawater dissolution rate of initial detonation agent Mercury Fulminate**

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**Abstract**

Mercury fulminate (HgFu) was used as an initial detonator for World War I and II munitions. Its presence in previously discarded and dumped munitions could potentially supply mercury pollution into coastal ecosystems where bygone weaponries reside. There is evidence that historical munitions have already contributed mercury pollution in coastal environments, and millions remain embedded in sediments and subjected to further weakening via corrosion under environmental conditions. Experiments were undertaken assessing HgFu dissolution under varying temperature and salinity conditions to constrain controls on mercury release into marine environments. Our results show that HgFu discharge is strongly temperature dependent, with dissolution rate constants increasing from ~0.4 mg cm-2 d-1 at 5oC to ~2.7 mg cm-2 d-1 at 30oC. No significant differences were observed between freshwater and seawaters up to 36 psu, except at 5oC. These experiments provide a basis for modeling HgFu release from underwater munitions and its dynamics in coastal environments.

**Keywords:** Mercury fulminate, munitions, dissolution rates, temperature, salinity

**Introduction**

Mercury fulminate (HgFu; Hg(CNO)2) was discovered by alchemists in the second half of the seventeenth century, likely by treating mercury (Hg) with nitric acid and alcohol under the ancient quest to transform Hg into gold. The devised HgFu crystals are stable, non-corrosive, and endothermic, but also highly responsive to friction, heat and shock. The original alchemists had no purpose for the resulting toxic and explosive material, but it eventually found broad use in military applications, and became a preferred primary explosive for fuzes in ancient and modern weaponry.

Alfred Nobel was credited as being the first to put HgFu into blasting caps for dynamite detonation, which in turn contributed to its widespread use in mining, demolition and eventually as an ignition trigger for bombs. Munitions including anti-aircraft grenades, mines and torpedoes, were used extensively during World War I (WWI) and World War II (WWII). During this period in the early 20th century the annual production of HgFu in Germany reached ~100,000 kg during the war periods (Kurzer, 2000; Matyas and Pachman, 2013). Some 20-30% of the munitions deployed during the WWs did not explode (Demining, 2016). Shipwrecks with munition cargo, and intentional jettisoning of munitions during wartime further added unexploded ordnance (UXO) to coastal waters. Moreover, after the surrender of the Axis powers concluding WWII, large quantities of munitions were deposited in the coastal North and Baltic Seas during and after the war period. Many of these munitions contained HgFu as the initial ignition agent in addition to the main charge of secondary explosives such as trinitrotoluene (TNT). In recent years there has been growing interest in the environmental impacts of legacy warfare chemicals and compounds, including Hg, TNT and other munition-related chemicals (Carton and Jagusiewicz, 2009; HELCOM, 2013; Beck et al., 2018; Kampmeier et al., 2020). Mercury fulminate in munition detonators is of particular concern given the high toxicity and mobility of Hg. Due to this effect, HgFu has largely not been applied as a primer since around the 1950s, and replaced by less detrimental and more efficient alternatives such as lead azide. Reportedly China and Russia could still be using HgFu in some function (Matyas and Pachman, 2013), thus its incidence has not completely disappeared. Dissolution rate is the initial control on release of HgFu into marine environments. Physical factors such as temperature and salinity can vary widely in munition-occupied coastal waters and likely influence HgFu stability and dissolution rates. To our knowledge dissolution rates of HgFu in seawater have not previously been investigated.

Mercury is a noxious, yet naturally occurring element, and is prominently released from geological reservoirs such as volcanoes and hydrothermal vents (Nriagu and Becker, 2003; Edwards et al., 2021). The amount of Hg input into the natural environment has furthermore increased significantly through anthropogenic practices, including gold mining, and the combustion of coal and other fossil fuels (Lamborg et al., 2002; Obrist et al., 2018; Outridge et al., 2018). Mercury is moreover transformed into methylmercury in aquatic environments, which is a prominent neurotoxin and health concern for humans and wildlife (Ullrich et al., 2001; Beckers and Rinklebe, 2017; Obrist et al., 2018). Therefore, it is important to trace and recognize latent point sources of Hg, such as the HgFu used in munition detonators, and its potential pathway into marine environments.

The properties which contribute to the functional success of HgFu also make it dangerous to prepare and investigate. For example, whilst HgFu has been known about and used for centuries, its chemical structure was only confirmed 300 years after its creation (Beck et al., 2007). By irradiating HgFu crystals with X-rays it was possible to corroborate that each Hg atom is surrounded by 2 carbon atom complexes, according a stretched out, nearly linear, orthorhombic molecular structure of O−N≡C−Hg−C≡N−O (Beck et al., 2007). Within this simple structure bond distances were determined as 2.029(6) Å for Hg−C, 1.143(8) Å for C≡N, and 1.248(6) Å for N−O. The structure angles are 179.7(6)o for C≡N−O, 169.1(5)o for Hg−C≡N, and a level 180(1)o for the central C−Hg−C (Beck et al., 2007).

Pure HgFu is a photosensitive substance which decomposes rapidly under UV light (Matyas and Pachman, 2013). Mercury fulminate has a density of 4.467 g cm-3 (Beck et al., 2007) and temperature dependent water solubility (Matyas and Pachman, 2013). There is a low explosion risk of HgFu at typical storage temperatures (Garner and Hailes, 1933; Bartlett et al., 1956; Matyas and Pachman, 2013). Decomposition of HgFu proceeds slowly (>20 months) and without explosive feedback up to 85oC (Ingraham, 1929; Narayana, 1944; Bartlett et al., 1956; Fendoroff et al., 1960; Matyas and Pachman, 2013). Increasing temperatures cause a volatile reaction to occur faster, and auto-ignition is at 170oC (Narayana, 1944; Bartlett et al., 1956; Fendoroff et al., 1960; Matyas and Pachman, 2013). Accordingly, experiments with HgFu need to be undertaken carefully and in a highly controlled manner in order to not accidentally trigger an explosive response.

Decomposition reactions of HgFu likely produce some combination of Hg salts and either carbon dioxide (CO2) or carbon monoxide (CO) and nitrogen (N2) gasses [1, 2]. Deterioration can also produce Hg(II) cyanate [3], an isomer of HgFu, thus yielding an identical chemical formula with a different atomic arrangement (Garner and Hailes, 1933; Bartlett et al., 1956; Matyas and Pachman, 2013).

[1] 4 Hg(CNO)2 → 2 CO2 + N2 + HgO + 3 Hg(OCN)CN

[2] 2 Hg(CNO)2 → 2 CO2 + N2 + Hg + Hg(CN)2 (cyanide)

[3] Hg(CNO)2 → :Hg(CNO)2 (cyanate)

[4] Hg(CNO)2 → 2 CO + N2 + Hg(0)

Mercury fulminate can disintegrate to yield elemental Hg (Hg(0)) [4] (Matyas and Pachman, 2013), or react and form fulminates with several other metals, which can then trigger further decomposition products. Mercury fulminate reacts violently under all conditions with aluminum and magnesium, while zinc and copper ensue a slower reaction (Fendoroff et al., 1960; Matyas and Pachman, 2013). Any Hg(0) ensuing from HgFu decomposition can also form amalgams with brass (a copper and zinc alloy), which in turn corrodes nearby metals. Brass and other metal alloys are typically used as ammunition cartridges or military casings, therefore any initial HgFu decomposition could lead to additional weakening of neighboring bomb exteriors. The aim of this study was to access the dissolution of HgFu in seawaters under varying conditions of temperature and salinity. The obtained data can thus be used for modeling of release and distribution of Hg from relict munitions in coastal waters.

**Methods**

Mercury fulminate was synthesized as described in Tomlinson et al (1971) at Fraunhofer Institute for Chemical Technology (Fraunhofer ICT). The HgFu product was in the form of brown-colored crystals, which is consistent with a highly pure composition (>99.5%) with no metallic Hg (Matyas and Pachman, 2013). Detonation sensitivity was tested according to DIN EN 13631-3 and DIN EN 13631-4, and indicated an impact sensitivity of 4 Nm with a friction sensitivity of <5 N.

The HgFu dissolution was determined at 6 to 24-hour intervals under a range of salinity and temperature conditions. All incubations were performed in artificial seawater made according to the recipe of Sunda et al. (2005). The primary solution was 36 psu, and intermediate salinities (0.1, 9, 18, 27 psu) were prepared via dilution with ultrapure water (MilliQ, Millipore, 18.2 MΩ cm). Experimental temperatures were held to 5, 20 and 30oC using stirred water baths. The initial incubation concentration comprised approximately 20 mg of HgFu placed in 100 mL of solution (200 mg/L). Complete dissolution was observed in several treatments within 24 h, and these treatments were repeated at a higher HgFu:solution ratio of 100 mg per 100 mL (1 g/L). However, given the limited available mass of HgFu, not all treatments could be repeated. All incubation treatments were performed in triplicate in acid cleaned borosilicate glass bottles. Samples (0.5 ml) were collected at five intervals over a 60-h period, and acidified to 4% (20 µl) with ultrapure HCl (Romil, UPA). Dissolved HgFu was measured as total Hg by high resolution inductively coupled plasma mass spectrometry (HR-ICP-MS; Element XR Thermofisher) at GEOMAR after a 1000 to 10.000-fold dilution with 1 M nitric acid (Romil, SPA, distilled). Analytical uncertainties were 1.2 ± 0.6% relative standard deviation (RSD) and 1.5 ± 0.5% for low- and high- solid experiments, respectively. Blank Hg concentrations were <1% of those in the samples.

**Results & Discussion**

The HgFu product had distinctive individual crystals which were approximately 100-300 µm in length (Figure 1 top). Dry HgFu reacts immediately and rapidly in the presence of low amounts of moisture (Matyas and Pachman, 2013). Whilst pure HgFu is classified as slightly soluble (Matyas and Pachman, 2013), our results indicated that HgFu dissolved readily in fresh and seawater solutions. Dissolution rates of HgFu were variable depending on temperature and salinity. A plot displaying typical dissolution data for three conditions is shown in Figure 1 (bottom).



**Figure 1**. Light microscope image of synthesized mercury fulminate (HgFu) crystals at 500 µm (top). Examples of dissolved mercury concentrations over the experiment incubation period for 3 different temperature treatments are displayed in the bottom plot. Regression lines indicate the linear region of the data used to calculate dissolution rates. Data points used for regression were the first two, four, then all six for 30, 20 and 5oC, respectively.

All treatments show that dissolved Hg concentrations increase rapidly during initial time points, until concentrations leveled after reaching complete dissolution. In effect, the dissolving amounts seem limited by HgFu mass availability in each treatment, and the plateaus represent a source exhaustion rather than saturation. Traditional dissolution models such as the stagnant film model could not be applied because saturation was not achieved. Therefore, dissolution rates were calculated from the slope of a linear best fit to the pre-plateau data (Figure 1 bottom). The immediate and sharp concentration increase is likely due to instantaneous dissolution of the finest HgFu crystals or fragments. This rapid dissolution was assumed to be an experimental artefact not representative of the true dissolution rate, thus the intercept of best fit lines was not considered further.

In the experiments with 200 mg/L HgFu (i.e. low-solids) maximum concentrations indicated complete dissolution of the solid material within 24 h, except those at 5oC, where dissolution was between 60 and 95%. Treatments which yielded especially swift dissolution (30oC at salinity 0.1 and 36 psu, and 20oC at 36 psu) were repeated using the higher 1g/L HgFu concentrations (i.e. high-solids). In high-solid experiments only ~50% of the solid HgFu was dissolved over the incubation period. Flocculation was observed in both high-solids treatments at 30oC, but not the 20oC treatment. There is no clear explanation for why this occurred, however it is possibly related to an aggregation of rapidly released Hg salts.

Dissolution rates (mg d-1) were normalized to the added solid HgFu surface area, which was estimated using HgFu density and average rhomboid crystal dimensions which were assessed from light microscopy images (i.e. Figure 1 top). Low-solids experiment had 20.3 ± 1.0 mg per incubation bottle, representing 8.2 ± 0.4 cm2 total surface area. High-solid experiments contained 102 ± 5 mg per incubation bottle, yielding 41 ± 2 cm2 total surface area. Dissolution rates ranged between 0.38 and 2.96 mg cm-2 d-1 for low-solid experiments, and between 1.52 and 2.65 mg cm-2 d-1 in high-solid experiments (Table 1). Uncertainty of the average rate based on triplicate incubations in each low-solid treatment was generally between 2 and 10% RSD, and between >8% up to 19% RDS for 30oC incubations. High-solid experiments had consistently higher RSD, between 19 and 31%, although absolute dissolution rates were similar to those in low-solid experiments. The reason for this higher variability is unclear. However, it’s possible that the flocculation observed within these treatments created interference towards dissolution physics and Hg concentrations, and hence consistency.

**Table 1.** Average (± SD) HgFu dissolution rates calculated for each salinity and temperature treatment. Low-solids and high-solid labels indicate incubations with 20 and 100 mg HgFu, respectively.

|  |  |  |
| --- | --- | --- |
| Temp (oC) | Salinity (psu) | Dissolution rate (mg cm-2 d-1) |
|  |  | ***Low-solids*** |
| *5* | *0.1* | 0.38 | ± | 0.03 |
| *5* | *18* | 0.55 | ± | 0.01 |
| *5* | *36* | 0.66 | ± | 0.03 |
| *20* | *0.1* | 0.91 | ± | 0.02 |
| *20* | *9* | 0.98 | ± | 0.07 |
| *20* | *18* | 0.97 | ± | 0.02 |
| *20* | *27* | 0.95 | ± | 0.05 |
| *20* | *36* | 0.88 | ± | 0.06 |
| *30* | *0.1* | 2.70 | ± | 0.54 |
| *30* | *18* | 2.55 | ± | 0.26 |
| *30* | *36* | 2.96 | ± | 0.23 |
|  |  | ***High-solids*** |
| *20* | *36* | 1.95 | ± | 0.62 |
| *30* | *0.1* | 1.82 | ± | 0.34 |
| *30* | *36* | 2.12 | ± | 0.45 |

Dissolution rates increased markedly with increasing temperature, with averages across salinities ranging from 0.52 ± 0.14 mg cm-2 d-1 at 5oC, then 0.93 ± 0.04 mg cm-2 d-1 at 20oC, to 2.73 ± 0.21 mg cm-2 d-1 at 30oC (Figure 2a; Table 1). A significant effect of salinity on dissolution rate was only observed in the 5oC incubations (Figure 2b), increasing from 0.38 ± 0.03 mg cm-2 d-1 (0.1 psu), then up to 0.55 ± 0.01 mg cm-2 d-1 (18 psu) and 0.66 ± 0.03 mg cm-2 d-1 at 36 psu. This suggests that temperature is the dominant factor towards dissolution rates, and salinity affects only become observable once temperature is low enough to exhibit less influence.

To hypothetically assess how HgFu dissolution out of a general UXO could be impacted by temperature and salinity, the dissolution of 3 different masses of HgFu were calculated for time increments (~0.1 - 0.5 day) up to 2 days using selected rates from Table 1. The amounts of HgFu gauged consisted of 30 and 300 mg HgFu, representing the typical range for a German shell (Command of the Army Council, 1943). Various larger WWII munitions were reported to contain up to 5.1 g HgFu (Command of the Army Council, 1943), therefore this was also considered.

Likewise, at a salinity of 0.1 psu approximately 2.2% of HgFu mass would dissolve within the first ~1.5 h (0.1 d) at 5oC, and increase to 3.8% HgFu lost at 36 psu. Spent mass per ~1.5 h would rise slightly to 5% at 20oC, but more than triple to 16.9% for 30oC at 36 psu. If salinity decreased to 18 psu, then the lost amount fell slightly to 14.6% at 30oC. Theoretically after 1 day ~38% of the original mass would have dissolved at 5oC (at 36 psu), compared to 50.3% at 20oC (SI; Table S1). Whereas all the original mass would have dispersed into the surrounding environment for all salinities at 30oC. Heavy-solid rates were also compared for 5.1 g of HgFu, and all yielded ~12% of HgFu mass dissolved after 1.5 h, and complete dissolution after 2 days. Consequently, once HgFu comes into contact with waters of various salinities and temperatures it will rapidly be transferred from solid crystals into surrounding waters.



**Figure 2.** Mercury fulminate dissolution rates as a function of (a) temperature and (b) salinity. Dark filled symbols marked with an asterisk (\*) indicate data from the high-solids incubations. Data points show the average rate from triplicate incubation vessels, with standard deviation displayed as error bars. Error bars not visible are smaller than symbol size.

These dissolution experiments were conducted using pure HgFu. However, HgFu in ignition fuzes from WWI and WWII typically consisted of HgFu blended with other ingredients and binders, such as antimony (III) sulfide (Sb2S3) and potassium chlorate (KClO3). It is unknown how these mixtures might influence HgFu dissolution, and experiments assessing all potential combinations were too complex and hazardous to perform. However, it is still important to note that differences in dissolution rates might vary for munitions residing in the environment due to these integrations. Composites were used to influence the physics of the desired detonation. For instance, the addition of KClO3 creates a slightly more brisant reaction than HgFu alone (Matyas and Pachman, 2013). The blends varied depending on the type of ammunition, and were moreover altered as detonators and ignitors are designed for completely different functions and purposes. Ignition propellants act to produce hot gas in order to accelerate the bullet, and likewise must not detonate as that would destroy the weapon. Accordingly, the propellent reaction should consist of a burst of hot gas and particles but no shock, while reactions for high explosives (e.g. mines) offer maximum shock. For example; grenade detonators would typically have 35.2% KClO3, 30.6% HgFu, 27% Sb2S3, with the remaining ~7.2% made of glass (Command of the Army Council, 1943). In contrast, pistol muniton was predominantly KClO3 (41.5%) and Sb2S3 (35.5%) and contained 23% HgFu (Command of the Army Council, 1943). The blends and binder proportions also varied depending on the manufacturing country and type of munition, as well as the explosive material used (Command of the Army Council, 1943; Matyas and Pachman, 2013). Likewise, it’s possible that variable proportions of each composite (e.g. greater amounts of KClO3) possibly assist with promoting HgFu dissolution. As a result, each piece and type of ammunition would carry a slightly different burden of chemicals, though ultimately still be lethal and dangerous.

As an example of how HgFu from WW residue munitions might contribute to the Hg load in a coastal environment, we estimated the Hg burden of the former Danish Flak training center Dänisch Nienhof (DN) located in Kiel Bay, along the Baltic coast of northern Germany. Roughly 1.5 million anti-aircraft grenades (Flak) were fired over a 10 km2 area near DN between 1939 and 1945 (MELUR, 2015). Approximately 2 g HgFu is expected to be in each flak grenade (MELUR, 2015). An estimated 70% of these grenades detonated, thus dispersing a total of ~2 t elemental Hg over the sea area (MELUR, 2015). Theoretically much of this Hg would became deposited in underlying sediments, with the remaining 30% (~1 t) inhabited within unexploded grenades dispersed throughout the DN area.

As follow up to this estimate, differences measured in sediments between DN and control sites outside of munition impacted areas were evaluated. Mercury concentrations for sediments in the vicinity of DN ranged from 48 up to 810 ng g-1 Hg (Gosnell, 2021). Considering that the area impacted by Flak at DN is 10 km2 (MELUR, 2015), and the typical bulk density of the sediment is 1.6 g c-3, this would result in an estimated range of 77 – 1300 kg Hg (~0.08 – 1.3 t) in surface sediments (the top 0.1 m) around DN. For comparison, sediment concentrations at nearby control sites range around 2 – 110 ng g-1 Hg (Gosnell, 2021), thus a 10 km2 area contains ~3 – 176 kg Hg (~0.003 – 0.18 t). As a result, the load at DN is enhanced by between 0.07 and 1.12 t of Hg compared to regional background levels. Therefore, it is possible an up to 7 times higher sediment Hg burden could be due to detonated and deteriorating WW munitions containing HgFu.

Any HgFu still remaining in WWII munitions would be rapidly transferred into the surrounding ecosystem upon degradation of munition casings. Considering the prevalent detection of chemical signatures such as TNT within coastal environments where munitions reside (Beck et al., 2018), it is likely that many have been and are in the process of disintegrating. Sediments can act as a sink for Hg (Ullrich et al., 2001), while Hg within the water column is more mobile and transitional, and can subsequently be circulated or purged into the atmosphere. Notably, as evident by the high Hg concentrations in sediments around munition impacted areas (Beldowski et al., 2019), it is feasible that HgFu dissolution has contributed some legacy pollution to the Baltic Sea. Climate change is projected to increase water temperatures, and nearshore environments where munitions reside are especially sensitive to warming effects (Friedland et al., 2012; Meier et al., 2022). In effect bottom water temperatures in the Baltic proper have reportedly been increasing by ~0.16oC year-1 (Rak and Wieczorek, 2012). Rising temperatures are further suggested to accelerate the breakdown of munition casings (Scharsack et al., 2021), and our experimental results demonstrate that increasing temperatures also cause HgFu to dissolve faster into surrounding waters. Climate change is additionally projected to bring a strong freshening of seawaters due to changes in hydrological cycling (Friedland et al., 2012). However, salinity yielded inconsistent influence on HgFu dissolution rates, therefore a freshening of resident seawater will likely have less of an impact towards HgFu degradation originating from munitions in the sea. The HgFu rate constant more than doubled when the temperature rose from 20 to 30oC, yet a freshening from 36 psu down to 0.1 psu deferred only a minor change (Table 1).

**Conclusion**

Millions of tons of relic munitions are present on the seafloor and represent a potential source of Hg to the environment. It is likely that nearly all of the WWII munitions containing HgFu have been subject to some deterioration, as all are now over 75 years old. The current results indicate that HgFu would readily dissolve into surrounding waters and porewaters once munitions casings are breached by corrosion and HgFu becomes exposed to ambient seawater. This would occur under typical temperature and salinity conditions for the North Sea and the Baltic Sea (i.e. 20oC and 18 psu), where many WWII munitions reside. The Hg released from damaged or corroded shells is likely to have been transferred into local environments and potentially sequestered in underlying sediments, harboring the WWs remnant ever longer. Furthermore, the degradation of munitions coinciding with increasing seawater temperatures will continue to release Hg into impacted regions as long as they reside within the brackish waters. These embedded munitions consequently provide an overtly targeted pollution source, and the eminent release of HgFu impends on any commitment towards general efforts to reduce anthropogenic Hg into the environment.

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