Metal spikes for incinerator and BIF compliance test and trial burn

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Abstract

This paper presents a discussion of the various chemical and physical forms of metal compounds that may be used for spiking during the trial burn or compliance test for hazardous waste combustion systems. It discusses the factors which should be considered in selecting the forms of the spiking metals for organic (hot, high Btu, or high heating value) waste streams, aqueous waste streams, and solid waste streams. The paper focuses on the organic waste streams and compares the use of organic metal dispersion to the use of organometal compounds or aqueous solutions of metal compounds as spikes for these types of feed streams. It is concluded that metal dispersions appear to form particulate which is in the appropriate micron range to tax the air pollution control system's performance. Dispersions of all regulated metals are commercially available in the required quantities for each application and they are relatively easy to pump, and to meter. Dispersions can be formulated so that their heating values are high enough to maintain the required elevated combustion and flame temperatures, and they are representative of the most common types of metal-bearing wastes sent to incinerators. The dispersions can also be used to spike metal compounds into water-based waste streams.

1. Introduction

The regulations for hazardous waste combustion in boilers and industrial furnaces (BIFs) [1, 2] limit the release of regulated metals on the basis of risk assessment procedures. The procedures were further explained in a series of policy statement [3] by EPA. While the original regulations were written only for BIFs permit writers have been advised by EPA management to also regulate metal emissions from hazardous

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waste incinerators under the 'omnibus' provision of RCRA and to evaluate the impact of each hazardous waste combustor, whether incinerator or BIF, through a site specific risk assessment based on the anticipated metals and organic emissions. It becomes increasingly necessary for operators of hazardous waste combustion equipment to consider spiking metal compounds during the trial burn or compliance tests. While the specific metals that need to be spiked for such testing can be readily determined, it is often difficult to establish the exact chemical form of the metals used for the spiking. It is required that all waste streams that may contain the metal be spiked during the test. As will be discussed below, the spiking of organic waste streams can prove difficult.

This paper focuses on the form of metal used to spike organic or high heating value waste streams. Specifically, it compares spiking organic waste streams with organometal compounds to spiking them with commercially prepared metal dispersions. The discussion is based on the basic principle described in both the Methods Manual [4] and the Technical Implementation Document [5] for the BIF Regulations that the test conditions, including the chemical and physical form of the spiking metals, should be that which is most likely to generate a fine particulate or fume. The concept of fume is not defined by these guidance documents, but the implicit definition is that a fume is particulate whose size range will tax the performance of the air pollution control device. This paper examines the use of metal dispersions as spiking material during the trial burn or compliance test and assess how they can produce the desired 'worst-case' conditions for the system.

It is recognized that compliance tests and trial burns are performed to achieve different regulatory goals. For the purpose of this paper the difference is unimportant and the terms will be used interchangeably.

2. Regulatory background

The metal emission limits are health, rather than technology based. The regulations specify the allowable ambient concentrations for the metals at a series of receptors around the site. Maximum acceptable emission limits are then determined for the facility using either site specific dispersion modeling or a general, highly conservative, dispersion model [4]. The acceptable emission limits are then used to set maximum allowable feedrate limits for the permit by one of the procedures described in the Technical Implementation Document [5]. The applicant can use three different approaches (termed tiers) for demonstrating compliance with the risk-based metal emission limits. The concept behind the tiers is that the closer to 'worst-case' the assumptions the applicant makes to estimate the metals emissions from the furnace, the less calculation or measurement is required.

Table 1 summarizes the assumptions made under each of the three (or four if Adjusted Tier 1 is considered to be separate) tiers. In order to understand the concept behind the 'tiers' it is necessary to recognize that the actual permit limit is based on a maximum acceptable ambient air concentration for the metal but the permit limit is for the maximum allowable metals feedrate to the furnace. The maximum allowable

Table 1
Determining allowable metals feedrates

Tier	Partitioning	Site specific disp. model
Tier 1	No	No
Adj. Tier 1	No	Yes
Tier 2	Yes	No
Tier 3	Yes	Yes

metals feedrate is related to the maximum allowable ambient concentration by two factors, the partitioning of each metal, and by dispersion.

Partitioning is defined (by EPA) as that fraction of the metal fed to the combustor which remains in the ash. This value is combined with the air pollution control system's (APCS) capture efficiency to determine the fraction of the metal fed to the combustor which is discharged from the stack. The trial burn establishes the minimum combined value of the combustor's partitioning and the APCS's capture efficiency. Since it is impossible to emit more metals from the stack than is fed to the combustor the assumption that all of the metal fed to the combustor is emitted from the stack is clearly worst-case and setting maximum allowable feedrate for the metal equal to its maximum allowable emission rate then satisfies all permit requirements without requiring emission testing for that metal. This assumption forms the first half of the Tier 1 or Adjusted Tier 1 permitting approach and will typically result in very low allowable metal feedrates as permit limits. It is up to the operator to determine whether these limits meet his or her needs.

It is also possible to make highly conservative assumptions about dispersion from the stack. The BIF regulations include tables which can be used in lieu of site-specific dispersion modeling. Once again, basing the allowable emissions on the look-up tables rather than on site-specific dispersion modeling should result in lower allowable metals feedrate limits. As shown in Table 1, the different tiers allow one to make one or both of these simplifying, but conservative, assumptions and to reduce the amount of testing or modeling required. While the three tier compliance approach is still in the regulations, it is not clear whether Tier 1 or Tier 2 still satisfy the present requirement for site-specific risk assessment. Consultation with regulatory authorities is highly recommended.

Inherent in the metals regulations is the assumption that the waste stream in which the metals are fed into the combustor will influence that metals emissions. Metals in the solid waste stream can be captured by the ash (partitioning), metals in a liquid waste stream are less likely to be captured by the ash and, hence, they are more likely to be vaporized or entrained and be emitted as a particulate. The permit conditions that result from such a test reflects this hierarchy. They are set on (a) total metals to the organic (high heating value) waste stream, (b) all liquid wastes to the combustor and (c) all wastes to the combustor. Clearly, if regulated metals will ever arise in the organic waste stream, they must be spiked into this stream during the test. If only the solid waste stream to the primary combustion chamber is spiked with a given

regulated metal during the test, the resulting permit condition would not allow any of that metal in any of the liquid waste streams. Similarly if only the aqueous waste stream is spiked, then the resulting permit condition will not allow metals in the organic waste streams [5].

3. Trial burn/compliance test conditions – definition of worst-case

If the conservative Tier 1 or Adjusted Tier 1 assumption (all of a metal fed to the combustor is emitted from the stack) do not meet the system's operating requirements, the owner/operator of a facility can usually establish higher feedrates by performing a compliance test or trial burn under worst-case operating conditions for the combustor. This test must demonstrate that the metal's emission rate at the desired metal feedrate is still below the allowable limit. The test measures the metal partitioning and removed in the APCS.

Worst-case operating conditions for the test refer to furnace and air pollution control equipment operation under conditions where the maximum metal emission are expected to occur. The concept is discussed in other papers [6–8]. Briefly, worst-case conditions require that the combustor be operated during the test at its maximum temperature, that the metals' feedrates in each waste stream (solid and liquid to each combustion chamber) are a maximum, and that the metals are fed into every waste stream that is likely to contain that metal during operation. The present discussion only deals with the comparative worst-case nature of different spiking forms of the metals. Factors such as quantity of metal fed, or overall furnace temperature can usually be maintained at a given level irrespective of the form of the spiking metal they will not be discussed further herein. For comparative purposes the highest fraction of the metal of interest will be emitted from the stack when: (i) the flame and combustion chamber temperatures are at or near their maximum, (ii) the waste is fed to the combustor in a form which reduces its likelihood of being trapped in the ash, and (iii) the resulting metal particulate is as small (fine particulate) as would likely occur in normal operation to provide a worst-case challenge to the air pollution control system.

The Guidance [5] on metals spiking is based on the assumption that combustion of the metal compound (i.e. of an organometal) and vaporization of the metal followed by condensation are the two dominant mechanism for forming fine particulate in the combustion chamber and thereby taxing the downstream air pollution control device. As such, it recommends that whenever possible the most volatile form of that metal (usually the chloride) or an organic compound of the metal be used as the spiking material. Let us examine these two assumptions starting with volatilization/condensation.

Clearly, temperature is a factor in fume formation. The trial burn is, thus, run at the maximum combustion chamber temperature but this is not necessarily sufficient. Worst-case conditions should also consider flame temperature for those metals which are fed in a high heating value waste. In past tests, aqueous solutions of the spiking metal compounds have been injected into the combustor as a side stream to the

organic waste feed. Some metal compounds, especially those of lead, are only moderately soluble in water so feeding the required amounts of metals, requires the injection of substantial amounts of water into the flame. The water creates cold spots in the flame, reducing the 'worst-case' nature of the test. In extreme conditions such injection can cause localized flame quenching and flame instability and make it difficult to maintain a maximum temperature in the combustion chamber. This type of spiking is also not representative of common furnace operation in many cases. In most furnaces, aqueous solutions of metal salts are fed into the combustor directly, and not fed through the same gun as the fuel. The most common types of metal-bearing organic waste is not an aqueous solution of metal salts, rather it is a suspension such as paint sludge.

The metal compound's vapor pressure or decomposition temperature also influences its volatilization. The higher the compound's vapor pressure the more the compound is likely to vaporize. Consideration of the heat and mass transfers involved in the volatilization lead one to conclude that the degree of volatilization will probably not increase significantly once the temperature exceeds the compounds boiling point. Above the compound's boiling point, the rate of volatilization will be limited by the rate of heat transfer into the metal-bearing particles and this will not be appreciably different for different forms of the metal if the particles are already in the low-micron size range. In other words, the rate of heat transfer into a particle of waste entering the flame zone will be more a function of the particle's size than of its composition.

The second mechanism for forming fine particulate is combustion. The Guidance [5] suggests that organic compound of the metal be used because it is a molecular phenomenon and, hence it is most likely to result in a fume of the metal. Other metal compounds than the organo-metals also burn, metal sulfides, for example, would also be expected to burn. Furthermore, other metal compounds can also participate in chemical reactions in the furnace and form small, possibly molecular sized particles. For example, carbonates and hydrates decompose and upon decomposition, they will tend to fragment into smaller particulate. In summary, many compounds of many of the regulated metals will form fine particulate which will tax the performance of the APCE.

4. Alternate metal spiking materials

The question then becomes, which compound of each regulated metal will result in the release of the largest amount of fine particulate during the trial burn. Three types of streams need to be spiked: (1) solid and non-pumpable sludge wastes, (2) pumpable aqueous wastes, and (3) non-aqueous organic wastes. The first two categories of waste are relatively simple to spike. Solid wastes can be spiked with powdered forms of the metal compounds. Aqueous wastes can be spiked with an aqueous solution of the metal compound. While this is consistent with policy, several points must be considered when preparing aqueous spiking solutions.

First is the solubility of each of the metal compounds. Some compounds, such as chlorides or nitrates of lead are relatively insoluble and may require relatively large

amounts of water to assure stable solutions. Second is the interaction of the various spiking compounds. Even though each compound is below saturation in a liquid the solubility product of the ion mix may result in precipitation of some metals. For example, if chloride salts of a number of metals are used to prepare the spiking solution, the overall solubility product for one or more of the metals may exceed saturation. The third issue is the chemical stability of certain forms of the metal. The most noteworthy is the stability of hexavalent chromium. Hansen [9] has shown that the aqueous solutions of Cr^{6+} compounds are highly unstable in the presence of even trace amounts of organic compounds. The regulations and guidance require that hexavalent chromium will be present in the spike whenever it may be present in the waste.

As discussed above, the spiking of metals into organic waste streams is often the most difficult. Many common compounds of the desired metals are insoluble in organic liquids. The BIF Guidance Document [5] suggests using organic organo-metal compounds or alternatives. Organo-metal compound spikes are consistent with the purpose of the test, but their use suffers from several practical problems. Most organo-metals are very expensive and often difficult or impossible to obtain in sufficient quantity. The BIF Guidance Document acknowledges this fact (pp. 5–9) and acknowledges expense or availability as a consideration. Organometallic compounds are also among the most toxic compounds known. They are not commonly used in commerce because of their toxicity and when they are used it is under tightly controlled conditions or in low concentrations. Organometallic compounds are rarely found in wastes. Spiking with organometallic compounds is, therefore, not representative of normal operation.

The Guidance [5] does mention the use of alternate forms of the metals, among them the use of dispersions. The dispersions appear to overcome many of the above problems associated with organo-metal compounds or with injection of aqueous solutions into an organic waste stream. The dispersion appears to be more representative of the forms of the metal which are likely to be found in organic wastes. It is the authors' experience that the most common form of metal found in organic waste is paint manufacturing sludge. Commercially available metal dispersions are similar in chemical properties, heating value, and consistency to latex paint. The dispersions are relatively stable suspension which can be readily pumped and accurately metered into the combustor during the trial burn or compliance test. If they are protected from freezing, the dispersions are stable for several months. The composition of the dispersion may vary somewhat depending on the specific metals blend desired. Typically, the dispersion can be made in either a pure oil or in an oil–water emulsion. A typically composition of the two types of dispersion is as follows. composition of aqueous metal suspensions: spiking metal compounds – 35%; oil – 41%; water – 12%; other ash (nonhazardous) 12%; composition of non-aqueous metal suspensions: spiking metal compounds – 40%; oil – 60%; other ash (nonhazardous) – 10%.

The ultimate analysis (wet basis) of the dispersion containing water is as follows, note that the ash includes the spiking metal compounds: C – 35.9; H (non-aqueous) – 5.1; Ash – 47.0%; H_2O – 12.0%.

The dispersions can be prepared from a wide variety of metal compounds; however the compounds must have a number of properties in order to form a stable suspension. First, the metal compounds used for the dispersion must be friable so they can be ground to a micron or sub-micron size. Second, the metal compounds used in the aqueous metal dispersions must be relatively insoluble in water and (for both dispersions) in fuel oil. Table 2 lists a number of compounds that have been successfully used to make stable suspensions. Table 2 also presents the melting, boiling and vaporization temperature for the metals and many of the compounds. The boiling and vaporization temperatures were obtained from the cited references; where indicated by the letter 'e', vapor pressure data from Barin [10] and from Hodgeman [11] were extrapolated using the following simplified Clausius–Clapeyron equation:

$$\ln(VP) = A + B/T,$$

where VP is the vapor pressure (Torr), A, B are the constants determined by regression from the available data, and T is the Temperature (K).

Vaporization temperature is defined as that temperature where the compound's vapor pressure equals 10^{-6} atm. The value has been used to estimate the likelihood of fume formation by different compounds of a metal [12, 13].

The dispersions appear to satisfy the requirements for meeting reasonable worst-case operating conditions for the combustor. Consider temperature. Injection of metal dispersions result in little or no reduction in flame or combustion chamber temperature. Adiabatic flame calculations (shown below) indicate that metal compound dispersions usually produce a flame which for all practical purposes is as hot as or hotter than that produced by organometallic compounds. The dispersion is made of finely ground (between 0.1 and 5 μM in diameter) metal compounds in a homogenized matrix of fuel oil and water. It is commercially prepared to a constant higher heating value (HHV) of approximately 8000 Btu/lb so it will sustain combustion and maintain flame and combustion chamber temperatures. The fine particle size also assures excellent heat transfer to the metal to encourage volatilization and/or any chemical changes that might occur in the combustor.

The adiabatic flame temperature calculations were performed for a typical high-heating value waste stream used for a trial burn with the composition shown in Table 3. The flame temperature was calculated for the pure 'waste' (20% excess air) and for a range of aqueous metal dispersion to 'waste' ratios. For comparison, the same adiabatic flame temperature was calculated for a mixture of 10% tetraethyl lead and 90% 'waste'. The results are given in Table 4. The difference in flame temperature for these various scenarios is well within that would be expected by variations in the normal composition of fuel oil and other constituents. Clearly the use of metal dispersion does not materially change the flame temperature. In all cases, the flame temperature is close to the volatilization temperatures for the metals in question and in most cases, it is above the boiling point or decomposition temperature of the metal compound. One would expect even less difference for the flame temperature when non-aqueous metal dispersions are used.

Next consider the likelihood of vaporization at combustor conditions. As shown in Table 2, most of the metal compounds which can be used in dispersions either have

Table 2
Compounds suitable for incorporation into stable oil dispersion

Metal/compound	Melting point (°C)	Boiling point (°C)	Vaporization temperature (°C)
Antimony metal		1635	≈ 550
Antimony oxide, Sb_2O_3 (Sb white)	655	1425	≈ 700
Antimony sulphide, Sb_2S_5 (Sb red)	d		
Antimony oxychloride, SbOCl	170		
Antimony trisulphide, Sb_2S_3	546		
Arsenic metal	814 +	615 s	
Arsenic oxide, As_2O_3 (white As)	307 s	193 s	
Arsenic disulphide, As_2S_2 (ruby As)		565	
Barium metal	725	1622	
Barium sulfate, $BaSO_4$ (blanc fixe)	1580	1149	
Barium hydroxide, $Ba(OH)_2 \cdot H_2O$	408	780	
Barium sulfite, $BaSO_3$	d		
Beryllium metal	≈ 1278	2467	≈ 1000
Beryllium oxide, BeO (beryllia)	257	≈ 3900	
Beryllium hydroxide, $Be(OH)_2$	138 d		
Beryllium acetate, $Be_4O(C_2H_3O_2)_6$	286	300 d	
Cadmium metal	321	767	
Cadmium selenide, CdSe (Cd red)	1350		
Cadmium sulphide, CdS (Cd yellow)	980 s		
Cadmium oxide, CdO (amorphous)	900 d		
Chromium metal	1890	2480	
Chromium VI compounds			
Strontium chromate, $SrCrO_4$			
(strontium yellow)	800		
Chromous chloride, $CrCl_2$	824		
Chromium III compounds			
Chromium oxide, Cr_2O_3			
(green cinnabar)	2435		
Chromic fluoride, $CrF_3 \cdot 4H_2O$	1000		
Chromic phosphate, $CrPO_4 \cdot 4H_2O$	d		
Lead metal	175	2026	
Lead oxide, Pb_3O_4 (red lead)	530	≈ 2000	
Lead monoxide, PbO (litharge)	888	1579 e	
Basic lead carbonate,			
$2PbCO_3 \cdot Pb(OH)_2$	400 d		
Mercury metal	− 39	357	
Mercuric oxide, HgO (red mercury)	d		
Silver metal		961	1950
Silver oxide, Ag_2O (argentous oxide)	300 d		
Silver sulfide, Ag_2S	825 d		

Table 2 Continued

Metal/compound	Melting point (°C)	Boiling point (°C)	Vaporization temperature (°C)
Thallium metal	302	1460	
Thallium sulfide, Tl_2S	443	d	
Nickel metal	1455	3187	1423 e
Nickel oxide, NiO (green)	1900		
Basic nickel carbonate, $NiCO_3 \cdot 2Ni(OH)_2 \cdot 4H_2O$	1450		
Nickel phosphate, $Ni_3(PO_4)_2 \cdot 7H_2O$			
Nickel hydroxide $Ni(OH)_2$	230 d		
Zinc metal	693	1184	
Zinc oxide, ZnO (chinese white)	1945	1800 s	
Zinc phosphate, $Zn_3(PO_4)_2 \cdot 4H_2O$	900		
Zinc sulfide, ZnS	1180 s		1400
Selenium metal	220	688	
Selenium disulfide, SeS_2	< 100	d	

Vaporization temperature: temperature at which the compounds vapor pressure equals 1 ppm (760×10^{-6} Torr); d: decomposes; s: sublimes; $\approx$: approximate, estimated; e: extrapolated from vapor pressure data; +: at 36 atm.

Data obtained from Barin [10] and Hodgeman et al. [11].

Table 3
Composition of trial burn high Btu 'waste'

C_6H_5Cl	13.0%
CCl_4	17.0%
$C_2Cl_3F_3$	1.2%
CH_3OH	44.4%
# 2 Fuel oil	21.4%
Sand/clay/ash	3.0%
HHV (BTU/lb)	9998

Table 4
Adiabatic flame temperature

% metal dispersion or tetraethyl lead	Adiabatic flame temperature (°F)
Pure waste	3618
1% Disp	3613
10% Disp	3569
10% TEL	3647

significant vapor pressures or they decompose at combustion temperatures. Most of the compounds boil at well below the flame temperature. As discussed above, if the compound boils or chemically decomposes at a temperature below that of the flame, its exact vapor pressure of the compound is unimportant. If the metal compound is a very fine particle (as it is in a dispersion), its propensity to vaporize and form a fume will be very high. As can be seen, many of the metal compounds that can be used in a dispersion have a boiling or vaporization temperature well below that of common flame temperatures.

The size of the metal compound particles in the dispersion itself lies in the range which taxes the performance of particulate emission control equipment even if no vaporization or other chemical change occurs. The metal compounds that are used for the dispersion are ground to create well-known particle sizes in the 0.1–5 μM size. This range could certainly qualify as a challenge to most types of air pollution control devices.

Because the metal compounds used must be friable in order for them to be successfully ground to this size range, they are susceptible to further size reduction by fragmentation when they hit the high temperature of the flame. They are also susceptible to size reduction by chemical changes. For example, the sulfides will burn, carbonates will roast, and metal hydrates will rapidly dehydrate. The release of carbon dioxide and water from the roasting and dehydration will fracture the particles even further. In short, one would expect the particulate produced by the combustion of metal dispersions to be at least as fine as those actually produced by the combustion of organometal compounds, and in many cases finer.

5. Conclusions

The criteria used for selecting the chemical form of metal compounds used for the trial burn are based on the theoretical behavior of that metal in the waste. Some factors which make the dispersion a good spiking material for metals trial burns:

(1) The small particle size of the metal compounds in the dispersion make them a challenge for all types of particulate air pollution control equipment even if further size reduction or volatilization were not to occur in the combustor.

(2) The small particle size allows maximum exposure of the metal compounds to flame temperatures. Since residence times in the flame are short, maximum exposure to the high flame temperatures encourages fume formation.

(3) The friable nature of the metal compounds used for the dispersion causes the particulate to fragment into even smaller particles upon exposure to flame temperature. The smaller particles further challenge the air pollution control device and encourage volatilization.

(4) Many of the metal compounds that can be used in a dispersion are volatile in their own right. Others, such as the sulfides of the metals burn and thus expose the metals on a molecular scale directly to flame temperatures. Other compounds are carbonates or hydrates which decompose at the conditions in the flame. The decomposition encourages fragmentation.

(5) The dispersion sustains combustion and thus intimately expose the metal compounds to flame temperatures. This encourages the sulfides to burn, volatile compounds to vaporize, and all particles to fragment.

(6) Cr^{6+} compounds remain stable in the dispersion until they hit the flame of the combustor posing a worst-case loading for this, regulated, form of chromium.

Based on these theoretical considerations, one can make equivalent arguments for metal dispersions and organometal compounds regarding the severity at which they test the combustor's air pollution control system during the trial burn. The dispersions offer a number of operating advantages over organo metal compounds for use in a trial burn or compliance test: ready availability; generally lower cost; excellent stability over time; easily pumped and metered with readily available equipment; lower toxicity; ease of handling; availability of a variety of controlled particle size distributions; wide selection of compounds which are compatible with the chemical processes within industrial furnaces; the physical properties of the material are very similar to paint and other metal sludges which are the most common forms of metal found in hazardous waste.

Because of these advantages, they appear to warrant serious consideration as metal spiking media for the high-heating value waste streams and, under certain conditions, for the solid waste stream during a trial burn or compliance test.

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