

AUTOIGNITION TEMPERATURE AS AN INDICATOR OF THERMAL OXIDATION STABILITY

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Summary

The concept and selection of Principal Organic Hazardous Constituents (POHCs) is an important part of the United States Environmental Protection Agency's hazardous waste incineration regulations. The POHC selection process is based on the concentration of organic constituents in the incineration waste feed and also on the degree of difficulty of incineration (thermal oxidation stability) of these constituents. As part of an EPA contract, nine potential thermal oxidation stability ranking parameters have been statistically evaluated for 15 compounds from existing vapor phase thermal oxidation data. Based on the evaluation, the autoignition temperature was found to be the best indicator of the degree of difficulty of incineration of the 15 compounds.

1. Introduction

In the June 24, 1982, *Federal Register* the EPA published the amended Phase II Incineration Regulations under the Resource Conservation and Recovery Act (RCRA) [1]. The concept and selection of Principal Organic Hazardous Constituents (POHCs) is an important part of these regulations. The POHCs, which must be proven during permit trial burns to be 99.99% destroyed or otherwise removed, are to be selected by the EPA permit writers from the listed Appendix VIII constituents present in the wastes. The POHC selection process will be based on the degree of difficulty of incineration (thermal oxidation stability) of the Appendix VIII constituents as well as on the concentration of Appendix VIII constituents in the incineration waste feed. Those Appendix VIII constituents present at significant concentrations and having the greatest degree of difficulty of incineration (highest thermal oxidation stability) will be the ones most likely to be designated as POHCs.

Appendix VIII is a list of about 370 organic and inorganic hazardous chemicals first published in Part 261 of the May 19, 1980, *Federal Register*. The Appendix VIII constituents have been defined by the EPA as substances which have been shown in scientific studies to have toxic, carcinogenic, mutagenic or teratogenic effects on humans or other life forms [2]. The

actual number of Appendix VIII chemicals is quite a bit higher than 370 because over 30 of the Appendix VIII constituents are general chemical groups such as isomers or salts. For example, the listed constituent "chlorinated benzenes" represents all the possible isomers of monochlorobenzene through hexachlorobenzene. The most recent version of Appendix VIII appears in the May 20, 1981, *Federal Register*.

As part of an EPA contract [3], various thermal oxidation stability (TOS) ranking parameters (TOSRP) have been evaluated from existing thermal oxidation data. Based on the evaluation, the autoignition temperature (AIT) was found to be a good indicator of TOS. This paper reports on the methods and results of this program to identify a suitable TOSRP.

2. Identification of a TOSRP

At the present time, very few incineration trial burn TOS data exist for the Appendix VIII constituents which can be used to help the permit writers select thermally stable POHCs. A sampling and analytical program using pilot- or full-scale incineration systems to determine thermal oxidation stabilities for the over 340 Appendix VIII organic constituents would be enormously expensive and time-consuming. As a substitute for a full-scale trial burn program, an accurate thermal oxidation stability ranking parameter (TOSRP) could be used to rank Appendix VIII constituents according to TOS in a less expensive and time-consuming manner.

Because of the large number of Appendix VIII hazardous constituents the ideal TOSRP should be simple, inexpensive to determine, obtainable in a reasonable time, and related to thermal oxidation destruction efficiency (DE).

The identification of a TOSRP required that a TOS database be collected from the literature for different chemicals. Laboratory-, pilot- and full-scale literature articles were reviewed, and TOS data were summarized when available. However, in order to compare and rank TOSs of a number of chemicals from different incineration systems the following factors must be similar: (1) degree of turbulent mixing; (2) sampling location relative to air pollution control equipment; (3) type of combustion, flame or flameless; and (4) incinerator outlet oxygen concentration. The only literature TOS data which matched these criteria for several different chemical constituents were from lab-scale vapor-phase thermal systems.

3. Literature review

The lab-, pilot- and full-scale incineration literature was evaluated for comparable TOS data based on the four factors previously listed. No full-scale thermal oxidation study was found in the literature which included data on the degree of turbulent mixing. Therefore, no TOS data from full-scale studies were used in developing the TOS ranking database.

The only existing pilot-scale study involving TOS data has been done by Bengt Ahling in Sweden. In a series of published articles [4–10] Ahling has studied the TOS of nine chemicals in a 6.9 GJ/h (6.5×10^6 Btu per hour) flame combustion incinerator. The Ahling data cannot be compared and ranked to lab-scale data because the lab-scale data are for flameless combustion. Thermal oxidation reaction rates in a flame zone are much faster than oxidation rates under flameless conditions because of the presence of high concentrations of free radicals in the flame zone [11].

Lab-scale TOS data that could be compared and ranked were obtained by the University of Dayton Research Institute (UDRI) [12–14] and the Union Carbide Corporation (UCC) [11, 15]. These studies involved flameless, well-mixed, vapor-phase thermal oxidation of various chemicals. Because the organic concentrations used were low, the thermal oxidation kinetics for the UDRI and UCC work was pseudo-first-order. Assuming first-order kinetics, the kinetic constants for 26 chemicals were calculated from UDRI and UCC DE data and are shown in Table 1. The T99.99/2, a UCC concept [15], is

TABLE 1

Summary of thermal oxidation kinetic parameters

Chemical	T99.99/2 (°C)	Collisional frequency factor, A	Activation energy, E_a (kJ g ⁻¹ mol ⁻¹)	AIT (°C)
Methane ^a	805	1.68×10^{11}	217.99	537
Decachlorobiphenyl	774	9.89×10^9	187.07	—
Hexachlorobenzene	767	4.16×10^{12}	238.10	—
2,2',4,4',5,5'-Hexachlorobiphenyl	752	3.48×10^{15}	292.03	—
2,2',4',5,5'-Pentachlorobiphenyl	736	5.42×10^{13}	252.53	—
2,2',5,5'-Tetrachlorobiphenyl	730	1.53×10^{14}	259.67	—
Vinyl chloride ^a	723	3.57×10^{14}	264.85	472
Benzene ^a	715	7.43×10^{21}	401.25	498
Dibenzofuran	715	8.70×10^{16}	307.98	—
Ethane ^a	714	5.65×10^{14}	266.10	472
Biphenyl	708	3.62×10^{15}	279.65	540
Dibenzo- <i>p</i> -dioxin	705	6.55×10^{18}	339.80	—
Propane ^a	704	5.25×10^{19}	356.48	450
Acrylonitrile ^a	703	2.13×10^{12}	217.99	481
Toluene ^a	700	2.28×10^{13}	236.40	482
Mirex	697	4.32×10^9	166.55	—
Ethylene ^a	695	1.37×10^{12}	212.55	450
Ethanol ^a	677	5.37×10^{11}	201.25	363
Propylene ^a	661	4.63×10^8	143.09	455
Butene ^a	642	3.74×10^{14}	243.51	384
1,2-Dichloroethane ^a	631	4.82×10^{11}	190.79	413
Ethyl acrylate ^a	588	2.19×10^{12}	192.46	273
Triethylamine ^a	567	8.10×10^{11}	180.75	232
Acrolein ^a	523	3.30×10^{10}	150.21	234
Kepone	473	8.99×10^{13}	189.82	—
DDT	447	2.94×10^7	93.76	—

^aChemical used in linear regression analysis.

estimated from these kinetic constants and is the estimated temperature necessary to produce a 99.99% destruction efficiency at a residence time of 2 s for a particular chemical in a well-mixed, flameless, lab-scale system.

The $T_{99.99/2}$ is estimated using the following first-order reaction rate kinetic equations:

$$k = \frac{\ln(C/C_0)}{t} \quad (1)$$

where k = reaction rate constant; C_0 = initial concentration of reactant; C = concentration of reactant at time t ; and t = time. The temperature dependence of the reaction rate constant k can be estimated by the well known Arrhenius equation [16] shown below:

$$k = A \exp(-E_a/RT) \quad (2)$$

where k = reaction rate constant (s^{-1}); A = collisional frequency factor (s^{-1}); E_a = activation energy ($kJ\ g^{-1}\ mol^{-1}$); R = universal gas constant ($0.008314\ kJ\ g^{-1}\ mol^{-1}\ K^{-1}$); and T = absolute temperature (K).

At a destruction efficiency of 99.99% and a residence time of 2 s, eqn. (1) reduces to:

$$k = - \frac{\ln[(1-0.9999)/1.0]}{2} = 4.6052 \quad (3)$$

Substituting this value of k into eqn. (2) yields the following expression for the temperature required for 99.99% destruction at a 2-second residence time ($T_{99.99/2}$):

$$T_{99.99/2} = \frac{-E_a}{\ln(4.6052/A) R} \quad (4)$$

Substitution of the kinetic constants A and E_a from Table 1 into eqn. (4) results in the $T_{99.99/2}$ value in kelvins shown in Table 1 converted to degrees centigrade.

The higher the $T_{99.99/2}$ value, the more stable is the chemical to thermal oxidation. The $T_{99.99/2}$ is an important parameter because it can be used to compare and rank TOS on a consistent basis.

4. TOSRP selection

The purpose of a thermal oxidation stability ranking parameter (TOSRP) is to provide a relative index of the thermal oxidation stability of various organic chemicals. An effective TOSRP must meet several criteria. The parameter should be highly correlated with TOS data, and must be either readily available in published reference books, easily derived from data published in the technical literature, or quickly and inexpensively obtained by laboratory testing procedures.

An effective TOSRP is one which correlates well statistically with the

$T_{99.99/2}$ value. Because of the absence of lab-, pilot-, or full-scale flame combustion $T_{99.99/2}$ data, the TOSRP at this time must be developed from non-flame laboratory premixed vapor-phase thermal oxidation data.

5. TOSRP results

Linear regression analysis was used to produce mathematical equations relating TOS, as $T_{99.99/2}$, to various kinetic constants, thermodynamic properties and physical properties. The parameters selected for evaluation were the following: autoignition temperature, ionization potential, heat of ion formation, molar heat of combustion, activation energy, heat of combustion, flash point, heat of formation at 298°C, and free energy at 298°C. The 15 compounds used in the linear regression analysis are from the UCC work [15] and are identified in Table 1.

Linear regression correlation coefficients (R) are presented in Table 2 for the correlation of the nine parameters. The correlation coefficients listed in Table 2 indicate that $T_{99.99/2}$ is highly correlated with autoignition temperature, with a correlation coefficient of 0.94.

Based on the close correlation between AIT and $T_{99.99/2}$ and the relative ease and low expense of measuring AIT values, it was recommended to the EPA that the AIT be chosen as a TOSRP.

TABLE 2

Results of linear regression analysis for correlation of $T_{99.99/2}$

X parameter	Y parameter	Correlation coefficient, R
Autoignition temperature (°C)	$T_{99.99/2}$	0.94
Ionization potential (eV)	$T_{99.99/2}$	0.80
Heat of ion formation (kJ mol ⁻¹)	$T_{99.99/2}$	0.74
Molar heat of combustion (J mol ⁻¹)	$T_{99.99/2}$	0.62
Activation energy (kJ g ⁻¹ mol ⁻¹)	$T_{99.99/2}$	0.42
Heat of combustion (J g ⁻¹)	$T_{99.99/2}$	0.39
Flash point (°C)	$T_{99.99/2}$	0.22
Heat of formation at 298°C (kJ mol ⁻¹)	$T_{99.99/2}$	0.21
Free energy at 298°C (kJ mol ⁻¹)	$T_{99.99/2}$	0.08

6. Autoignition temperature

Autoignition temperature (AIT) has historically been used in the chemical industry as an indicator of the potential explosion hazards of chemicals. The AIT involves combustion in the absence of an ignition source and is the lowest temperature at which a material begins to self-heat at a high enough rate to result in combustion [17].

A connection between the AIT and TOS was indicated almost 10 years

ago in a paper on fume incineration of solvent-air mixtures [18]. In this paper an incineration temperature several hundred degrees fahrenheit above the solvent's AIT was recommended for solvent destruction. In a more recent reference [15], multiple regression techniques were used to develop predictive equations for the thermal destruction efficiencies of various chemicals in terms of AIT, molecular structure and incineration residence time. The AIT and residence time were found to be the two most significant variables in the predictive equations.

The AIT has been found to change substantially when measured using different experimental conditions. Some of the variables known to affect the AIT are the organic vapor and oxygen concentrations, the shape and

TABLE 3

Autoignition temperature values of Appendix VIII constituents^a

Chemical	AIT value (°C)	Chemical	AIT value (°C)
Phenol	715	Vinyl chloride	472
Dichloromethane	662	1,1-Dichloroethane	458
<i>o</i> -Dichlorobenzene	648	Vinylidene chloride	458
Chlorobenzene	638	1,1,2-Trichloroethene	457
Diphenylamine	634	Ethylene oxide	429
Chloromethane	632	Isobutyl alcohol	427
Hexachlorobutadiene	618	Formaldehyde	424
Aniline	615	Methyl methacrylate	421
Resorcinol	608	1,2-Dichloroethane	413
Cresylic acid	599	Epichlorohydrin	411
Benzyl chloride	585	Trichloroethylene	410
Phthalic anhydride	584	Di- <i>n</i> -butyl phthalate	403
1,2,4-Trichlorobenzene	571	Acetyl chloride	390
Acetophenone	570	Allyl alcohol	378
Cresol	559	Creosote	336
2-Chloronaphthalene	558	Ethyleneimine	322
1,2-Dichloropropane	557	Propyl amine	318
Dimethyl phthalate	556	1,2,3-Trichloropropane	304
Formic acid	539	Acrolein	278
Hydrocyanic acid	538	Nitroglycerin	270
2-Picoline	538	Hydrazine	270
Bromomethane	537	Hydrogen sulfide	260
Naphthalene	526	Nicotine	244
Acetonitrile	524	Paraldehyde	238
Acrylamide	524	Benzotrichloride	211
Methyl ethyl ketone	515	Crotonaldehyde	207
Benzene	498	Methyl hydrazine	194
1,1,1-Trichloroethane	486	Dimethyl sulfate	188
Nitrobenzene	482	Acetaldehyde	185
Toluene	482	1,4-Dioxane	180
Pyridine	482	Phosphine	100
Acrylonitrile	481	Carbon disulfide	90
Maleic anhydride	477		

^aAppendix VIII constituents from May 20, 1981, *Federal Register*.

volume of the experimental vessel, materials of construction of the experimental vessel, and the experimental pressure [19, 20]. Because of this experimental variability, if the AIT is to be used as a relative TOSRP, the experimental conditions for each chemical tested must be as close to identical as possible. Table 3 summarizes the published AIT values for Appendix VIII constituents. AIT values were found for only about 20% of the Appendix VIII constituents and these AIT values were not determined using the same experimental conditions. The lowest reported AIT values were used for the chemicals summarized in Tables 1 and 3.

7. Autoignition test

A standardized ASTM test exists for the determination of the AIT of liquid chemicals. This test method is performed in a heated 500-ml borosilicate, round-bottomed, short-necked boiling flask, using a 100- μ l or 100-mg sample. The sample is added in a darkened room to a preheated flask containing air at a specific temperature and ambient pressure, and observed for 10 minutes. The test is done repeatedly at different temperatures to obtain the lowest temperature at which a sudden appearance of flame and a sharp rise in gas temperature occur [21]. If no flame or sharp temperature rise occurs within 10 minutes, another temperature is tested. The AIT test is primarily for liquids but the method can also be used for solid chemicals which melt and vaporize or readily sublime at the test temperature. The test is not designed for chemicals that are solids or liquids at the test temperature, or are capable of exothermic decomposition at test temperatures [21]. The ASTM AIT test for liquid chemicals (ASTM E659-78) takes approximately 4–8 hours for the determination of an AIT value. The ASTM test is designed to obtain the minimum AIT based primarily on considerations of safety. It appears likely that the ASTM method could be modified to save experimental time and money by reducing or eliminating the AIT determinations done at different sample concentrations. This modification and others could result in a shorter, more economical test designed specifically for TOS ranking rather than safety considerations.

8. AIT data

Considering the unknown variability of the AIT data, verification of the existing Appendix VIII AITs using a consistent experimental procedure would be a valuable step towards confirmation of the AIT as a suitable TOSRP. In addition to unknown variability, some of the individual AIT values are suspect. For example, based on bond energies and other factors, it appears likely that the reported phenol AIT value of 715°C is higher than it should be.

AIT values can be obtained from Zabetakis [22], the Manufacturing Chemists Association [23], Harris [24], Kuchta [25], and the National

Fire Protection Association [26]. References [23] and [26] have the most extensive AIT data.

9. AIT data gaps

As can be seen from Table 3 chemicals without existing AIT data include highly chlorinated hydrocarbons, polycyclic aromatics, and substituted benzenes and phenols. Many of the highly chlorinated hydrocarbons are nonflammable under normal conditions of temperature, pressure and oxygen concentration and may not have been tested for AIT because these chemicals were not considered to present fire or explosion hazards. Carbon tetrachloride (CCl_4), for example, was used for many years as a fire-extinguishing fluid. Kirk-Othmer [27] states that the AIT of CCl_4 is above 1000°C , but gives no experimental reference for this statement. It is not known at this time whether AITs can be experimentally obtained for highly chlorinated hydrocarbons using an ASTM E659-78-type method.

As previously mentioned, ASTM E659-78 cannot be used to measure the AITs of chemicals that are solids or liquids at the test temperature (plastics, high-boiling-point chemicals) [21]. Methods are available, however, to measure the AIT of solids and/or high-boiling-point liquids [28, 29] but it is not known whether AITs determined by these methods would correlate as well with $T_{99.99/2}$.

If AITs cannot be obtained for highly chlorinated hydrocarbons and high-boiling liquids, TOSs can be determined by a UDRI-type laboratory-scale vapor-phase thermal oxidation system. The UDRI-type system will be an important part of a POHC research program necessary to rank Appendix VIII constituents according to thermal oxidation stability. The UDRI-type system can be used both to generate TOS data and to verify and further develop the validity of AIT and/or other parameters as TOSRPs.

10. Final comments

At the present time, because of the lack of $T_{99.99/2}$ data under flame combustion conditions, the validity of the AIT as a TOS indicator for real-world flame combustion incineration systems is still in question. Because of this and the small AIT/Appendix VIII database (about 60 AIT values), the EPA is using heat of combustion as a TOS indicator [30]. Even though the correlation of the $T_{99.99/2}$ data with heat of combustion is not as good as the AIT correlation (see Table 2), the heat of combustion database is large and good techniques are available for the estimation of heat of combustion.

In order to develop the TOSRP concept further, a great need exists for additional developmental work on AIT, heat of combustion and other potential TOSRPs. Additional work should be done in the areas of UDRI-type vapor-phase destruction efficiencies and lab- or pilot-scale flame combustion destruction efficiencies as well as development of a much greater, consistent AIT database.

Since the TOSRP correlation database is limited in number (15) and in type of molecular structure, it is recommended that UDRI-type thermal oxidation destruction data be developed for 15 to 30 more selected chemicals. The new UDRI-type DE data can be used to expand the AIT and TOSRP correlation database. The chemicals chosen for the UDRI-type experimental work should be selected from Appendix VIII, be present at high concentrations in high-volume industrial wastes, and come from other structural families than the 15 chemicals previously correlated.

Since the recommendation of the AIT as a TOSRP is based only on flameless premixed vapor-phase thermal oxidation data, a TOSRP research program is also required to further verify under flame combustion conditions the concept of the TOSRP. Such a program has been started and will include the following activities [31]:

(1) Laboratory thermal oxidation under flame conditions will be conducted on various Appendix VIII chemicals to determine if a statistically significant correlation exists between thermal destruction with a flame, the AIT, and other possible TOSRPs.

(2) The data generated in these lab thermal oxidation studies under flame combustion conditions will be compared to the flameless Dayton and Union Carbide vapor-phase laboratory data. If a good correlation exists between the flame combustion and flameless thermal oxidation data, the less expensive flameless lab-scale thermal destruction work could be used to expand the Appendix VIII thermal oxidation stability database.

The TOSRP shown in such a program to have the most significant statistical correlation with thermal oxidation stability under flame conditions should then be adopted by the EPA as the TOSRP. A POHC selection process by the EPA incineration permit writers which would incorporate the identified TOSRP would then be based on the most scientifically valid information available.

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