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Project Summary

Pilot-Scale Evaluation of the Thermal Stability POHC Incinerability Ranking

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A series of pilot-scale incineration tests was performed at the Environmental Protection Agency's (EPA's) Incineration Research Facility to evaluate the thermal-stability-based principal organic hazardous constituent (POHC) incinerability ranking. In the tests, mixtures of 12 POHCs with predicted incinerabilities spanning the range of most- to least-difficult-to-incinerate classes were combined with a clay-based sorbent and batch-fed to the facility's pilot-scale rotary kiln incinerator via a fiberpack drum ram feeder. Five tests were completed. Kiln operating conditions were varied to include a baseline operating condition, 3 modes of attempted incineration failure, and a worst-case combination of the 3 failure modes.

Kiln exit POHC destruction removal efficiencies (DREs) were in the 99.99% range for the volatile POHCs during the baseline, mixing failure (increased charge mass), and matrix failure (decreased feed H/Cl) tests. Semivolatile POHCs were not detected in the kiln exit for these tests; corresponding DREs were generally greater than 99.999%. The thermal failure (low kiln temperature) and worst-case (combination of thermal, mixing, and matrix failure) tests resulted in substantially decreased kiln exit POHC DREs. These ranged from 99% or less for Freon 113 to greater than 99.999% for the less-stable-ranked POHCs. General agreement between relative kiln exit POHC DRE and predicted incinerability class was observed for those two tests.

This Project Summary was developed by EPA's Risk Reduction Engineering Laboratory, Cincinnati, Ohio, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

One of the primary functions of the EPA's Incineration Research Facility (IRF) is to conduct research activities for the EPA's Office of Solid Waste (OSW) in support of regulation development and implementation. One major regulatory issue of high priority during 1990 concerned the evaluation of an incinerability ranking system for POHCs. The system was developed over the past several years by the University of Dayton Research Institute (UDRI) under contract to EPA's Risk Reduction Engineering Laboratory (RREL).

The 1981 hazardous waste incinerator regulations require that an incinerator undergo a trial burn performance test in order to become permitted to operate. This trial burn is required to show that the incinerator is capable of achieving the mandated 99.99% POHC DRE. In trial burn planning, the incinerator operator is required to select POHCs using two criteria: concentration in the waste and difficulty to thermally destroy, or "incinerability." The incinerability ranking included in the 1981 regulations was based on compound heat of combustion.

The heat of combustion ranking has several acknowledged deficiencies, however. Thus, EPA initiated studies to define or develop alternate, more suitable incinerability ranking approaches. One such approach is the thermal-stability-based POHC ranking, developed by UDRI. This ranking is based on the temperature required to achieve 99% destruction at 2 sec residence time under oxygen-starved conditions as measured in laboratory experiments. As of early 1990, the fundamental basis supporting the ranking approach had been documented and sufficient information to rank the organic hazardous constituents had been collected. Since it is based only on laboratory-scale data, evaluation of the thermal stability POHC incinerability ranking under actual incineration conditions became a high-priority research need for 1990.

The test program described in the full report was designed to develop the data to evaluate the POHC incinerability ranking at the pilot scale. The specific objective of the test program was to measure the DRE of a number of POHCs under each of several modes of incinerator operation, and compare relative POHC DREs as a function of incineration conditions and feed characteristics. The comparison would facilitate a determination of how relative POHC DREs compared with expectations based on the thermal stability ranking.

In the tests, a mixture of 12 POHCs with predicted incinerabilities spanning the range from the most-difficult-to-incinerate class to the least-difficult-to-incinerate class was tested. This "POHC soup" mixture was combined with a clay-based sorbent solid matrix and packaged into fiberpack drums for incineration testing in the rotary kiln incineration system (RKS) at the IRF. The drums containing the soup/clay mixture were batch fed to the RKS via a fiberpack drum ram feeder.

A series of five incineration tests was performed during which incinerator operating conditions and test mixture composition were varied. Specific test program variables were:

- Kiln temperature
- Feed batch charge mass
- Feed composition, specifically H/Cl ratio

One test was performed under typical operating conditions with a baseline mixture composition. The other tests varied the above in an attempt to simulate various modes of incineration failure, where incineration failure is defined to exist when POHC DREs are less than 99.99%. The various modes of incineration failure attempted were thermal failure, mixing failure, feed matrix effects, and a worst case combination of these.

Test Program

Test Facility

The IRF's RKS was used for this test program. A process schematic of the RKS is shown in Figure1. The IRF RKS consists of a primary combustion chamber, a transition section, and a fired afterburner chamber. After exiting the afterburner, flue gas flows through a quench section followed by a primary air pollution control system (APCS). The primary APCS for these tests consisted of a venturi scrubber followed by a packed-column scrubber. Downstream of the primary APCS, a backup secondary APCS, comprised of a demister, an activated-carbon adsorber, and a high-efficiency particulate (HEPA) filter, is in place.

Synthetic Waste Mixture

Twelve POHCs were selected for inclusion in the synthetic waste mixture employed in the test program. The incinerability ranking groups 333 POHCs included into 7 stability classes from most stable (class 1) to least stable (class 7). UDRI recommended that 2 compounds from each class be included in the test mixture and provided a list of candidates for selection. The selection of compounds from this candidate list was guided by sampling and analysis, compound compatibility, compound availability, and safety considerations.

The compounds selected for the test mixture are listed in Table 1. The table also notes the composition of two test mixtures containing the POHCs. Test mixture 1 was the baseline test mixture. The POHC concentrations in test mixture 2 represent adjustments to relative POHC concentrations to yield a mixture with decreased H/Cl ratio.

The mixtures incinerated in the test program were prepared using commercially-available pure chemicals and materials. Test material formulation consisted of adding weighed quantities (1.3 kg, 3 lb total) of the mixture of the 12 organic constituents to a weighed quantity (2.3 kg, 5 lb) of an absorbent clay. The clay/organic mixtures were packaged into 1.5-gal fiberpack drums lined with polypropylene bags, the mouths of which were closed with wire ties.

Test Conditions

The variables for the test program were the H/Cl ratio in the synthetic waste feed, kiln temperature, and synthetic waste feed charge mass. Five tests, specified to be conducted with various combinations of these parameters, were selected to evaluate the relative incinerability of the POHCs. The target test matrix is shown in Table 2. Test 1 represented a baseline, or normal, set of incinerator operating conditions. Test 2 attempted thermal failure by decreasing the kiln exit temperature to a target of 649°C (1200°F). To further promote thermal failure, 0.9 kg (2 lb) of water was added to each waste feed charge for Test-2. In Test 3, mixing failure was attempted by doubling the drum charge mass from 3.6 to 7.3 kg (8 to 16 lb). This doubled charge mass was introduced at half the baseline frequency, so as to maintain the overall waste feedrate equal to that for the other test conditions. Test 4 was designed to investigate the effects of reducing the H/Cl ratio (matrix failure) in the waste feed. Test 5 combined the three failure-promoting conditions to produce a "worst-case" condition by operating with the kiln exit temperature at a target of 649°C (1200°F), introducing the waste at double the baseline charge mass, and by using a low H/Cl ratio waste mixture.

For all tests, the average kiln exit temperatures were within 14°C (26°F) of the respective target temperatures. However, actual O_2 levels in the kiln exit flue gas were generally higher than the target concentrations. The higher O_2 levels were generally the result of higher than expected air in-leakage into the kiln chamber.

Sampling and Analysis Procedures

The scope of the sampling effort undertaken during this test program is illustrated in Figure 2, in which the sampling locations and the corresponding sample collection methods are identified. Specifically, the sampling effort during each test consisted of:

- Obtaining a sample of the POHC/clay feed mixture by compositing the contents of 3 waste fiberpack drums randomly selected during the test
- Obtaining a sample of the scrubber blowdown liquor composited from grab samples taken at hourly intervals over the test period
- Obtaining a sample of the kiln ash by compositing three samples from the ash collection bin at the end of the test
- Continuously measuring O_2, CO, CO_2, and unheated total unburned hydrocarbon (TUHC) concentrations in the flue gas at the kiln exit; O_2 concentrations at the afterburner exit; O_2, NO_x, unheated TUHC, and heated TUHC concentrations at the scrubber exit;

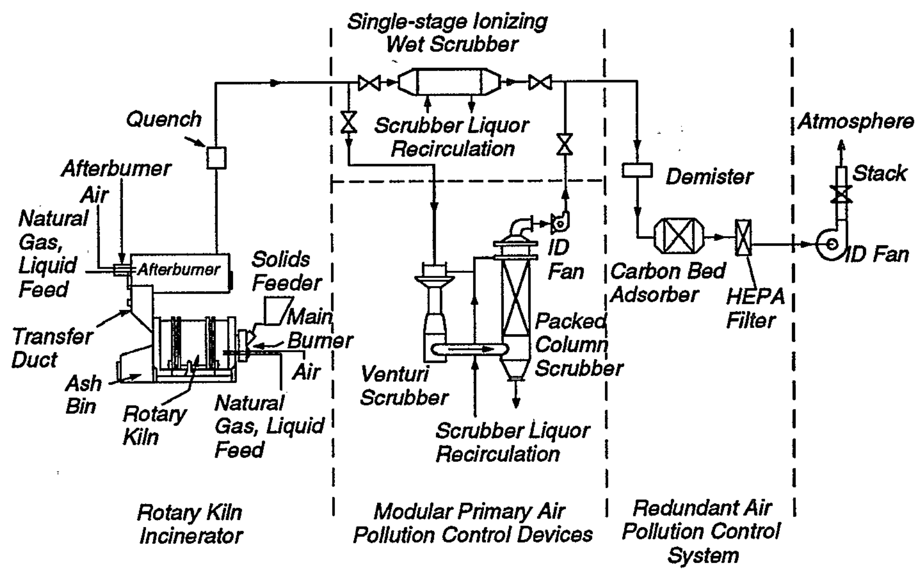


Figure 1. *Schematic of the IRF rotary kiln incineration system.*

Table 1. *Synthetic Waste Mixture Composition*

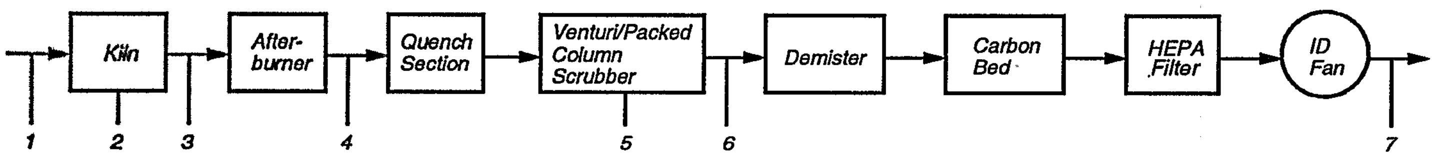
Component	Concentration (Wt %) Mixture 1 High H/Cl	Mixture 2 Low H/Cl	$T_{99}(2)^a$ (°C)	Rank[b]	Stability Class
Benzene	8	4	1,150	3	1
Chlorobenzene	8	4	990	22	1
Tetrachloroethene	8	33	890	43	2
1,2,2-Trichloro-1,1,2-Trifluoroethane (Freon 113)	8	4	780	92	3
Benzenethiol	8	4	725	122	3
Nitrobenzene	8	4	655	150/151	4
Hexachlorocyclohexane (Lindane)	10	5	645	159	4
Hexachloroethane	10	25	585	213	5
1,1,1-Trichloroethane	10	5	545	233	5
p-Dimethylaminoazobenzene (methyl yellow)	10	5	~400	268	6
Nicotine	10	5	<320	286 to 289	7
N-nitroso-di-n-butyl amine	2	2	<320	316 to 331	7
H/Cl (molar)	3.6	1.2			

[a] Temperature required to achieve 99 % destruction in 2 sec.
[b] Incinerability rank in list range from most refractory (No. l) to most labile (No. 333).

Table 2. Target Test Conditions

Test	Kiln H/Cl (molar)	Kiln exit temperature °C(°F)	Kiln exit O^2 (%)	Afterburner exit temperature °C(°F)	Afterburner exit O^2 (%)	Organic/clay per fiberpack kg (lb)	Feed regimen drums/charge	Charge hr
1	22.8	871 (1600)	10.4	982 (1800)	9.0	3.6 (8)	1	12
2	22.8	649 (1200)	12.6	982 (1800)	9.1	4.5 (10)[a]	1	12
3	22.8	871 (1600)	10.4	982(1800)	9.0	3.6 (8)	2	6
4	15.7	871 (1600)	10.4	982 (1800)	9.2	3.6 (8)	1	12
5	10.3	649 (1200)	13.2	982 (1800)	9.2	3.6 (8)	2	6

[a] 0.9 kg (2 lb) water added per fiberpack.



Sampling point	Sample Location: Feeds and residuals: POHC/ Clay Mixture	Kiln ash	Scrubber blowdown	Continuous monitors: O_2	CO_2 CO_2	NO_x	Unheated TUHC	Heated TUHC	Flue gas: Method 0010 (semivolatile organics)	Method 0030 (volatile organics	Method 5 (Particulate and HCl)
1	X										
2		X									
3				X	X		X	X	X	X	
4				X							
5			X								
6				X		X	X	X	X	X	
7				X	X			X	X	X	X

Figure 2. Sampling matrix.

and O_2, CO, CO_2, and heated TUHC concentrations in the stack

- Sampling flue gas at the kiln exit, scrubber exit, and stack for the semivolatile and volatile POHCs using Method 0010 and Method 0030, respectively

The laboratory analysis procedures used to characterize the samples collected over the test program included:

- Analyzing the composite feed, kiln ash, and scrubber blowdown samples from each test for volatile and semivolatile organic constituents
- Analyzing Method 0010 train samples from each test for semivolatile organic hazardous constituents
- Analyzing Method 0030 train samples from each test for volatile organic hazardous constituents

Semivolatile organic analyses were performed by Method 8270. Solid samples, including waste feed and kiln ash, and Method 0010 samples were Soxhlet-extracted by Method 3540 in preparation for analysis. Liquid samples were liquid-liquid extracted by Method 3510. Method 0030 (VOST) sample analysis was by thermal desorption purge and trap GC/MS (Method 5040) analysis with an ion trap detector.

Test Results

The POHC measurements at the kiln exit are the most relevant with respect to evaluating the incinerability ranking in that the incineration failure conditions tested involved varying kiln operation. Thus, incineration failures achieved would be most evident and best measured at the kiln exit.

Figures 3 through 7 show kiln exit POHC DREs measured in bar chart form. The POHCs are ordered along the horizontal axis by their thermal stability index ranking from predicted most-stable (benzene) to least-stable (N-nitroso-di-n-butyl amine). The vertical axis is the quantity [-log (1-DRE/100)] for each POHC, which represents the "number of 9's" of POHC destruction. A value of 1 signifies 90% DRE, a value of 2 signifies 99% DRE, and so on. Each bar represents the measured DRE for the corresponding POHC. Where flue gas analysis indicated that the particular POHC was below its detection limit, a stacked bar format is used to convey this information. The height of the bottom bar of the stack represents the DRE calculated using the practical quantitation limit (PQL). The combined stacked bar is extended to the top of the chart, as a visual reminder that the POHC was not detected and that the measured DRE was greater than that computed using the PQL.

The POHC DREs illustrated in Figures 3 through 7 were calculated using prepared synthetic waste feed formulation data. Feed samples were analyzed for each test, as noted above. However, analyzed POHC concentrations in feed samples were substantially lower than the concentrations corresponding to the POHC quantities used to form the POHC mixtures. On average, only between 12 and 29% of the volatile organic constituents and between 27 and 82% of the semivolatile organic constituents in the prepared mixtures could be accounted for in the feed analyses. Fiberpack weights measured during the tests rule out mass evaporative loss as the explanation for differences between prepared and analyzed concentrations. One possible explanation is that the organic liquid constituents were so tightly adsorbed to the porous clay that the sample preparation procedures associated with Methods 8240 and 8270 analyses could not quantitatively free the organic constituents for detection in the analyses. All test program data support this hypothesis and it is believed that what was prepared was indeed fed. For this reason, prepared-composition-based DREs alone are presented. Nevertheless, all conclusions regarding relative DREs are also supported using analyzed feed composition data.

Table 1 noted that benzenethiol was selected as one of the class 3 POHCs in the POHC mixture. However, while performing tests to verify that stable synthetic waste organic feed mixtures could be prepared, it was discovered that benzenethiol quite rapidly and completely reacts, in the presence of the other organics and the clay matrix, to form diphenyl disulfide, a class 6 compound. As a result, diphenyl disulfide, not benzenethiol, was actually fed to the incinerator. Thus, the DRE for diphenyl disulfide is shown in Figures 3 through 7 bar charts, and its bar location corresponds to its class 6 incinerability order.

No DREs for nicotine are shown in Figures 3 through 7. Nicotine recoveries from matrix spike Method 0010 samples were generally poor. These poor recoveries prevented making accurate assessments of the DREs for this compound and it has been omitted from the figures.

The following discusses relative POHC DREs measured for each test in turn.

Test 1—Baseline Incineration Conditions

The incinerator operating conditions for Test 1 represented baseline incineration operation, which, from past experience would result in acceptable POHC destruction. As shown in Figure 3, kiln exit DREs were 99.99% or greater for all POHCs. Benzene, chlorobenzene, tetrachloroethene, Freon 113, and 1,1,1-trichloroethane were quantitatively measured at the kiln exit and their corresponding DREs are shown by the single bars. The remaining POHCs were not detected at the kiln exit and their respective DREs were represented by the two-segment stacked bars, the significance of which was discussed above.

The high POHC DREs confirmed that this baseline incinerator operating condition was indeed capable of satisfactorily destroying even the predicted most difficult to incinerate POHC, benzene. A weak correlation might exist between DRE and the POHC incinerability ranking, in that, except for 1,1,1-trichloroethane which had a DRE of about 99.99%, DREs for POHCs ranked in class 4 and above were higher than the class 3 and below POHCs. It bears emphasis, however, that the measured POHC DREs only varied by small degree (from 99.994% to 99.9997+%). This, coupled with the lack of gross incineration failure to broaden the incinerability response, could explain the inability to establish a clear correlation between DRE and the incinerability ranking index from the baseline test data.

It is interesting to note that 1,1,1-trichloroethane, a POHC ranked in class 5 and believed to be relatively easy to incinerate, had a measured DRE substantially lower than similarly ranked POHCs. One possible explanation is that 1,1,1-trichloroethane is a common product of incomplete combustion (PIC), and can be formed during the incineration process, potentially from hexachloroethane, another component of the POHC mixture.

Test 2—Thermal Failure (Quenching)

Test 2 was intended to simulate a thermal failure condition through incineration quench. This was accomplished by lowering the kiln temperature from nominally 871°C (1600°F) to 649°C (1200°F) via 2 means. A measured amount of water contained in a polyethylene bag was added to each waste feed fiberpack drum and the kiln was fired at very high air/fuel ratio. These two actions in combination would be expected to create conditions conducive to the formation of cold POHC-containing pockets of gas which would escape the kiln prior to being destroyed.

Figure 4 presents the kiln-exit POHC DREs for this test. The data clearly indi-

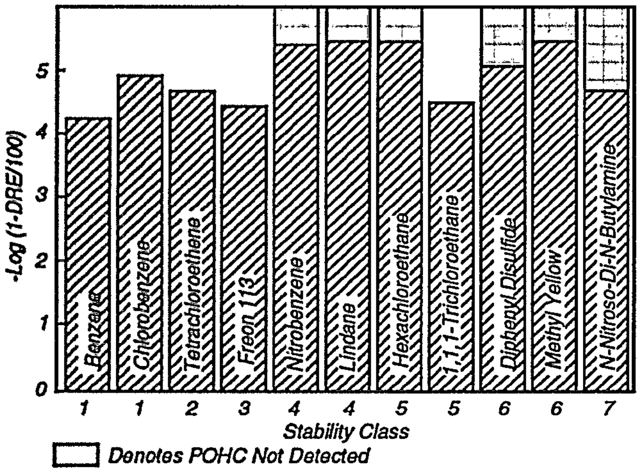


Figure 3. Kiln exit POHC DREs for Test 1.

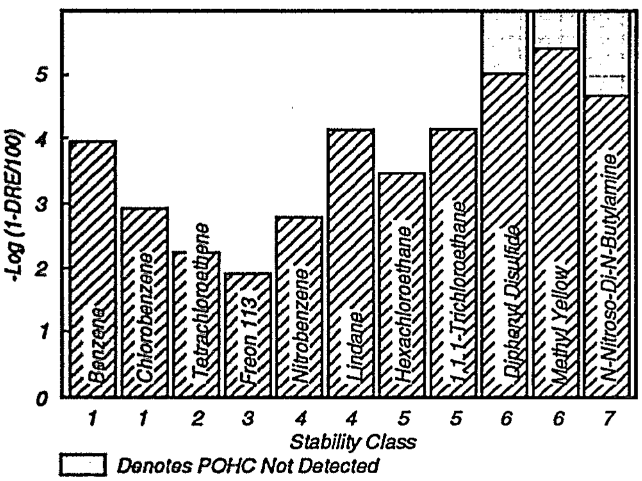


Figure 4. Kiln exit POHC DREs for Test 2.

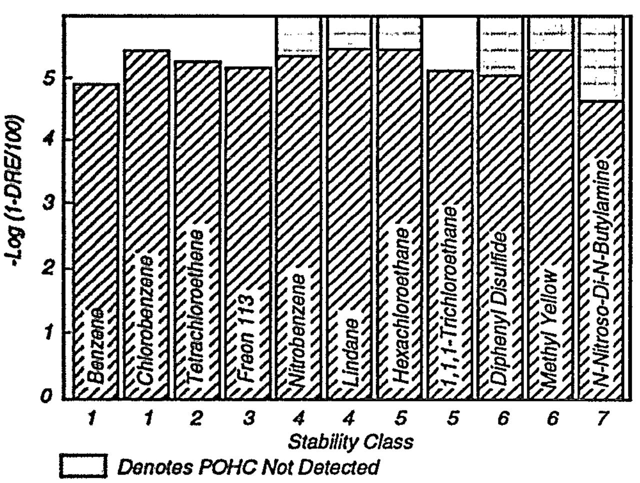


Figure 5. Kiln exit POHC DREs for Test 3.

cate that this test condition resulted in significantly different POHC DREs compared to the baseline test. A wide range of POHC DREs was observed, from less than 99% for Freon 113 to greater than about 99.999% for diphenyl disulfide, methyl yellow, and N-nitroso-di-n-butyl amine. The low DREs for several POHCs confirmed that incineration failure did occur during this test.

With the exception of a few anomalies (discussed below), a general correlation between DRE and incinerability ranking seems apparent for this test. The observed DREs for the class 3 to 7 POHCs appeared to follow the incinerability ranking predicted behavior. Some POHC-to-POHC variability existed within this sub-group of POHCs. Lindane exhibited a higher DRE than the neighboring ranked POHCs.

A considerably more significant deviation from incinerability ranking expectation involved the DREs of the 4 most-stable ranked POHCs within classes 1, 2 and 3. The relative DREs measured for these four POHCs were in an order opposite to the ranking predictions. The relative extent of incineration failure for these 4 POHCs was not in accordance with expectations from the thermal stability ranking.

Test 3 —Mixing Failure

One of the modes by which POHCs can escape an incinerator undestroyed results from the lack of adequate mixing between POHC and oxidizer. For Test 3, the weight of each waste charge to the kiln was doubled, while the hourly waste feedrate was maintained at a level consistent with the other four tests. The doubled waste charge was thought to increase the likelihood of creating oxygen-deficient pockets of POHCs in the kiln chamber. The expectation was that if the oxygen-deficient conditions persisted through the kiln, undestroyed POHCs could escape the kiln chamber.

However, as the data in Figure 5 show, no clear failure was apparent during this test. All POHC DREs were greater than 99.99% and exhibited trends similar to those observed for the baseline condition test (Test 1). DREs for nitrobenzene and the group of POHCs ranked easier to incinerate were high. Within this group, only 1,1,1-trichloroethane was detected in the kiln exit flue gas sample at a level above its PQL. The other less-stable ranked POHCs were not detected in the kiln exit flue gas.

The four most difficult to incinerate POHCs, benzene, chlorobenzene, tetrachloroethene, and Freon 113 were present in the kiln exit flue gas sample at

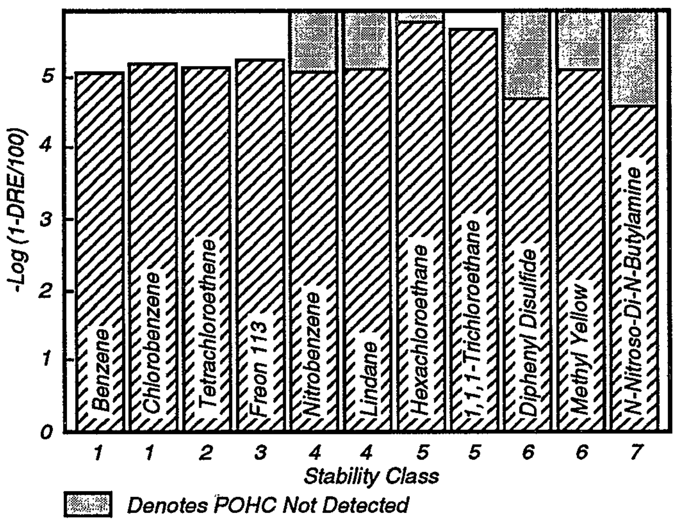


Figure 6. *Kiln exit POHC DREs for Test 4.*

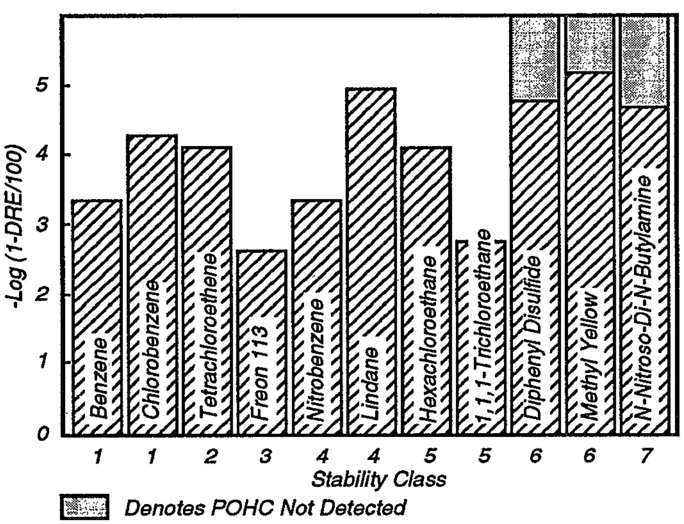


Figure 7. *Kiln exit POHC DREs for Test 5.*

levels corresponding to between 99.99 and 99.999% DRE.

No correlation between POHC DREs and POHC incinerability ranking was apparent for this test. DREs for the volatile POHCs (detected in the flue gas) were comparable to the DREs associated with the PQLs for the semivolatile POHCs (not detected in the flue gas).

Test 4—Matrix Failure

This test (Test 4) attempted to cause incineration failure by decreasing the H/Cl ratio in the organic feed to the kiln. The H/Cl ratio in the feed waste for Test 4 was 1.2, as compared to a H/Cl ratio of 3.6 for the baseline Test 1.

The kiln exit POHC DREs for this test are shown in Figure 6. These were uniformly high: all exceeded 99.99%. As in Test 3, no correlation between POHC DRE and incinerability ranking was apparent because the POHC DREs were uniformally high.

One possible explanation for the inability to achieve POHC DRE failure in this test is that the actual H/Cl ratio in the kiln environment as a whole was quite different from that in the waste feed itself. This was so because the auxiliary fuel for the burner, in this case natural gas, was a significant additional source of hydrogen. If this source of hydrogen is included, the H/Cl ratio in the total kiln environment for this test would be 15.7, which may be considerably higher than the H/Cl ratio required to cause DRE failure.

Test 5 —Worst Case Combination

This last test was conducted to present the most challenging combination of the mechanisms tested in terms of POHC destruction failure. The kiln was operated at the reduced temperature of 640°C (1184°F) to induce thermal failure; the waste feed charge size was doubled to promote mixing failure; and the chlorine content in the feed waste was elevated to promote matrix failure. It should be noted that, in a departure from Test 2 procedures, no water was added to the feed waste fiber drums, because doing so would introduce a quantity of hydrogen that might nullify any potential elevated chlorine (matrix failure) effect.

Figure 7 shows the POHC DREs for this test condition. In this test, 8 of the 11POHCs were detected in the kiln exit flue gas. This was in contrast to only five POHCs being found at concentrations above their PQLs during baseline Test 1. The 3 most easily predicted incinerated POHCs, namely, diphenyl disulfide, methyl yellow, and N-nitroso-di-n-butyl amine were not found above their PQLs. Assuming that these POHCs were present at their respective PQLs would lead to computed POHC DREs > 99.998% for these 3 POHCs. These DREs, being higher than those measured for the remaining POHCs, were consistent with their incinerability ranking indices.

The DREs for the 8 quantifiable POHCs ranged from over 99 to almost 99.999%, calculated based on feed formulation data. Lindane had the highest DRE at 99.9989%. Freon 113 and 1,1,1-trichloroethane exhibited the lowest DRE at about 99.8%. While no monotonic correlation between POHC DREs and incinerability ranking order existed for this test, a weak relationship may have existed for the class 3 through class 7 POHCs. As observed in Test 1, 1,1,1-trichloroethane exhibited a DRE significantly below that of its neighboring ranked POHCs.

It is interesting to note how well the relative POHC DREs of this "worst-case" test compared to those observed for Test-2. Recall that Test 2 simulated only quench failure. The relative DREs for these two tests exhibited similar patterns although 2 differences could be noted. One difference is the absolute DRE levels, which for this test were generally higher than those observed for Test 2. The other difference relates to the DREs for benzene and 1,1,1-trichloroethane. The DREs for benzene and 1,1,1-trichloroethane were nearly 2 "nines" higher for Test 2 than for Test 5.

Conclusions

Conclusions from the tests include the following:

- The baseline operation condition resulted in effective POHC destruction. Kiln exit POHC DREs were in the 99.99% range for the volatile POHCs in the test mixture. Semivolatile POHCs were not detected in the kiln exit flue gas; corresponding lower bound DREs were generally greater than 99.999%.
- Neither the mixing failure nor matrix failure attempts resulted in incineration failure. Kiln exit POHC DREs were comparable to those measured in the baseline test for all POHCs.
- The thermal failure and worst-case tests resulted in kiln POHC destruction failure. For both tests, kiln exit POHC DRE ranged from 99% or less for Freon 113 to greater than 99.999% for the highest-ranked (least-stable) POHCs.
- For the incineration failure tests, there was general agreement between observed relative kiln exit POHC DRE and thermal stability incinerability ranking expectations. However, two deviations occurred for both tests.
  - The class 1 compounds (benzene and chlorobenzene) and the class 2 compound (tetrachloroethene) were less stable (had greater kiln exit DRE) than the class 3 compound Freon 113.
  - 1,1,1-trichloroethane was apparently more stable in the baseline and worst-case tests than its class-5 ranking would suggest, when compared to the other class 5 and the class 4 compounds; production of 1,1,1-trichloroethane as a PIC could account for this observation.

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The complete report, entitled "Pilot-Scale Evaluation of the Thermal Stability POHC Incinerability Ranking," (Order No. PB92-166 966/AS; Cost: $35.00, subject to change) will be available only from:
National Technical Information Service
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