

Standardizing the Measurement of Minimum Extinguishing Concentrations of Gaseous Agents¹

by

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Abstract. The cup-burner has had a long history as a tool for measuring the minimum flame extinguishing concentrations of gaseous agents required for flammable and combustible liquids. There have been, however, material inconsistencies in reported values of minimum extinguishing concentrations of inert gas agents (Senecal, 2005). Analysis indicates that there is a basis to expect highly consistent, and even predictable, results. It appeared that the likely source of measurement variation was due to procedural and mechanical differences in the conduct of the cup-burner test among various laboratories. A Task Group, appointed by the NFPA GFE-AAA technical committee on gaseous fire extinguishing systems, revised the cup-burner procedure including the specification of a standardized test apparatus. This paper describes an inter-laboratory study conducted in 2006 to (1) determine consensus value benchmark minimum extinguishing concentrations (MEC) of an inert gas agent and a halocarbon agent; (2) evaluate the reproducibility of the revised test method; (3) evaluate the repeatability of the test method; and (4) to suggest how benchmark MEC values might be used to harmonize values reported by other laboratories. The results of an inter-laboratory (round robin) study have established benchmark MEC values for nitrogen and HFC-227ea with excellent reproducibility. The results also show that MEC values reported by most laboratories agree closely though there are cases where significant systematic differences in MEC were reported suggesting a need for review of procedural details. Additionally, an approach is suggested for applying benchmark extinguishing concentration data to harmonize future cup-burner measurements involving agents and fuels other than those used in the round robin study.

Introduction. Fire extinguishing systems employing gaseous agents are used widely to protect an array of hazards found in business, commercial and industrial settings. The workhorse gaseous fire extinguishing agent from the 1960's to the early 1990's was bromotrifluoromethane, better known as Halon 1301, a potent ozone depleting substance. Broad international endorsement of the Montreal Protocol on Substances that Deplete the Ozone Layer (1987) and, its subsequent amendments, has led to the near world wide cessation of production of Class I ozone depleting substances, including halons.² A number of alternative gaseous fire extinguishing agents have been successfully introduced into the marketplace to serve in the stead of Halon 1301. National and international standards have been developed to help manufacturers, distributors, installers and consumers of these products understand their effective and safe use.

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² See <http://www.epa.gov/ozone/ods.html> for a list of Class I ozone depleting substances.

The effectiveness of a gaseous fire extinguishing agent is determined by laboratory scale and large scale tests fire extinguishing tests. These tests are used to determine the minimum extinguishing concentrations (MEC) of an agent in air that achieves the specified extinguishing performance metric. Product approval procedures³ specify use of large scale tests to determine agent MECs required to extinguish flames of different fire threats consisting of Class A⁴ and a Class B fuels⁵. The cup-burner test is the one laboratory scale procedure for determining agent MEC for flammable or combustible liquids, i.e. Class B fuels. The cup-burner test method is described in NFPA and ISO standards on gaseous fire extinguishing systems (Annex B of NFPA 2001; Annex B of ISO 14520) but the test specifications differ in several details. The MEC of a gaseous agent is different for each fuel. The technical committees that developed the approval standards recognized that performing large scale fire extinguishing tests for every Class B fuel is impractical. Thus, both national and international standards on gaseous fire extinguishing systems require the use of the cup-burner method to establish MEC values of a Class B fuel in lieu of large scale pan-fire tests. The relevant standards language is as follows:

NFPA 2001: 5.4.2.1 The flame extinguishing concentration for Class B fuels shall be determined by the cup-burner method described in Annex B.

ISO 14520: 7.5.1.2 The minimum Class B design concentration for each extinguishant shall be a demonstrated extinguishing concentration for each Class B fuel plus a safety factor of 1.3. The extinguishing concentration used shall be that demonstrated by the cup-burner test, carried out in accordance with the method set out in Annex B, that has been verified with the heptane pan tests detailed in C.5.2. For hazards involving multiple fuels, the value for the fuel requiring the greatest design concentration shall be used. The extinguishing concentration shall be taken as the cup-burner value or the heptane pan test value (see Annex C), whichever is greater.

When the hazard being protected is a Class B fuel both the NFPA 2001 and ISO 14520 (and others) require that the minimum design concentration (MDC) of a gaseous agent be at least 1.3 times the agent's MEC for that fuel. Thus, the cup-burner test is thrust into a prominent role related to the specification of the MDC of gaseous agents for fire extinguishing systems. In order to well serve the stakeholders in the fire protection community the cup-burner test method must be both accurate and reproducible. Concerns related to the reproducibility of the cup-burner method, described previously [Senecal, 2005], led to an initiative by the NFPA technical committee responsible for gaseous agent standards. The technical committee appointed a Task Group to study and improve the cup-burner method with the specific goal of improving inter-laboratory reproducibility. In May 2006 the author presented the results of Task Group effort to date which was the development of a more highly specified test procedure and the design and distribution of a standardized test apparatus [Senecal, 2006]. Lacking at that time was data from an inter laboratory study, or "round robin" test program, that demonstrated the reproducibility of the revised cup-burner procedure and standardized test apparatus. An inter laboratory study was

³ See Underwriters Laboratory standards UL-2127 and UL-2166 as well as ISO standard ISO 14520.

⁴ The standard Class A fuel test articles include a wood crib and arrays of polymer sheets employing using three different types of plastics (polypropylene, PMMA – polymethylmethacrylate, and ABS – acrylonitrile butadiene styrene polymer), each article constructed and tested according to established specifications.

⁵ The large scale Class B fuel fire test consists of extinguishing a pan fire of the burning fuel. Pan sizes vary slightly among standards but are approximately 0.25 m² in area. Heptane is the most commonly used Class B reference fuel.

conducted during the summer of 2006. The results of that study, the subject of this paper, (a) show that the revised cup-burner test method employing a standardized test apparatus has very good reproducibility, and (b) establish benchmark extinguishing concentrations for two reference agents, a halocarbon agent and an inert gas agent. The benchmark extinguishing values, now established with a high level of confidence, can serve as a basis for standardization of extinguishing data for different gaseous agents and Class B fuels.

Inter-Laboratory Study. Nine laboratories acquired the new NFPA standard cup-burner and agreed to participate in the inter-laboratory study. The study had three objectives.

1. To establish that the revised test method and apparatus had achieved a high degree of reproducibility as evidenced by the ability of multiple laboratories to obtain MEC values that were statistically equivalent.
2. To propose that the new MEC values be adopted as industry benchmark data for industry use.
3. To reveal information about intra-laboratory repeatability and its implications, if any, about conducting tests used to establish MEC values for gaseous agents.

The inter-laboratory study requested that participants use the revised cup-burner test procedure as described in NFPA 2001 Annex B as approved through the ROC stage of the 2007 revision cycle.⁶ An important element of the study was the use of the new NFPA standardized cup-burner apparatus, shown in figures 1 and 2. The test elements included the following:

- Fuel: n-heptane
- Agent 1: Nitrogen, representing the properties of inert gas agents
- Agent 2: HFC-227ea, representing the properties of halocarbon agents
- No. trials: Three (3) trials, made on separate days, for each agent
- Same operator conducts all tests
- No. data points: Each trial is to consist of five (5) consecutive data points.

Reporting: Report trial date, barometric pressure, individual test results, trial average, trial standard deviation. Concentration to be reported in mol %⁷. A formatted Excel spreadsheet provided to standardize reporting.

Data analysis: Participants were asked to submit data to Fire Protection Research Foundation, One Batterymarch Park, Quincy, MA 02269-9101, Attn: Ms. Kathleen Almand, Executive Director.

Data reporting: The FPRF received all data, maintained confidentiality by assigning each lab a number, analyzed the data and reported the data to the NFPA cup-burner Task Group without reference to participating laboratories by name.

⁶ The Report on Comments (ROC) stage is the final approval step by an NFPA technical committee.

⁷ There appeared to be some inconsistency in the units reported, vol. % or mol %. There is no difference between the two for an ideal gas agent, such as nitrogen. There is a very small difference for a halocarbon agent such as HFC-227ea of the order of 1% of the reported value.

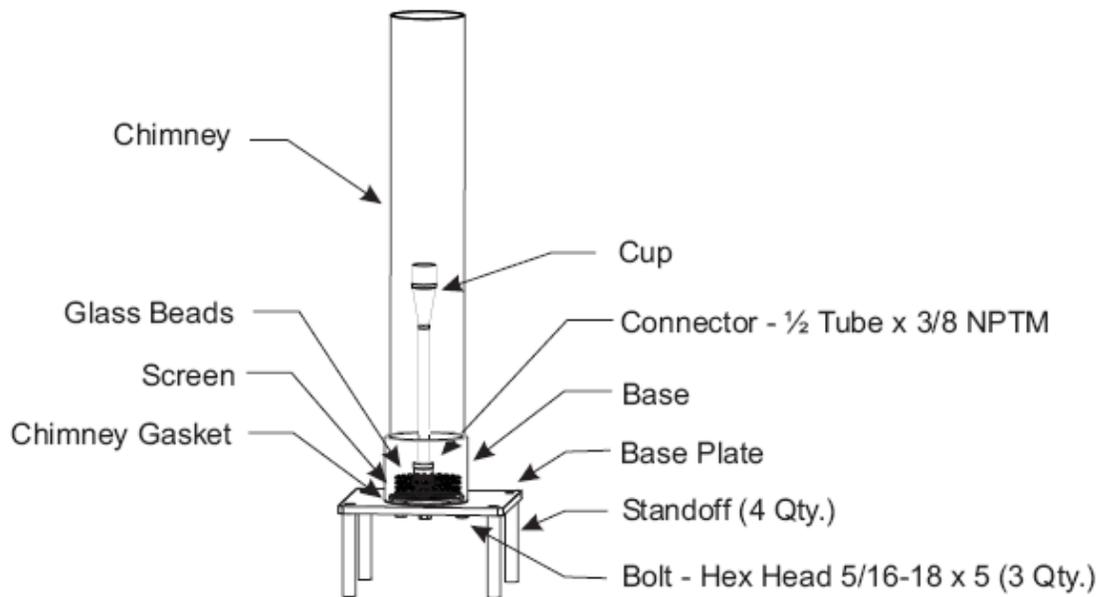


Figure 1. NFPA standard cup-burner assembly

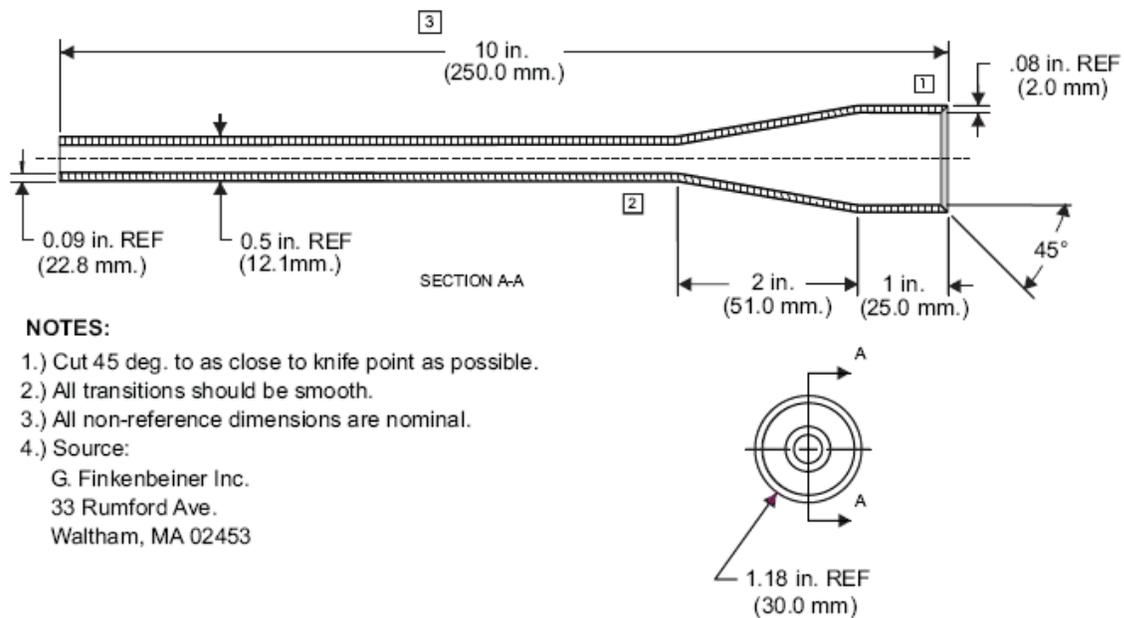


Figure 2. Cup details

Results. Not all participating labs tested both agents. The results are discussed in terms of both laboratory averages and day-to-day variations.

Nitrogen. Table 1 shows the test result set for the eight laboratories that participated in the nitrogen extinguishing tests. The daily averages are based on the five extinguishing values reported for the day. The standard deviation value for each lab was on all 15 reported data points.

Lab #	Day 1	Day 2	Day 3	3-day Average	3-day Range	St Dev*	Range/ St Dev
1	32.11%	32.37%	32.58%	32.35%	0.47%	1.13%	0.41
2	34.13%	32.12%	31.98%	32.74%	2.14%	1.26%	1.70
4	32.46%	32.76%	32.83%	32.68%	0.38%	0.62%	0.60
5	31.64%	31.86%	32.89%	32.13%	1.25%	0.59%	2.12
6	31.65%	30.19%	30.56%	30.80%	1.45%	0.80%	1.82
7	32.04%	32.14%	33.08%	32.42%	1.04%	0.72%	1.44
8	26.51%	26.51%	28.33%	27.11%	1.82%	1.00%	1.82
9	32.01%	32.30%	32.08%	32.13%	0.29%	0.38%	0.78

* Standard deviation based on all 15 reported measurements, five each on three days.

The three-day laboratory average results are shown graphically in figure 3. The results of six labs were in very close agreement and served as the basis of calculating the consensus average MEC for nitrogen as 32.44 vol % for n-heptane flames. The dotted lines represent the bounds of the six-lab average plus and minus one standard deviation of 0.85 vol % (2.6% of the consensus mean) which was calculated based on all 120 nitrogen data points submitted. These data serve to establish the position that the revised cup-burner test method and apparatus are fully capable of providing highly reproducible results for inert gas gaseous extinguishing agents.

The average results for Lab 6 was 30.8 vol % which is 1.9 standard deviations below the consensus mean.

The average results for Lab 8 was 27.1 vol % which is 6.3 standard deviations below the consensus mean.

Repeatability is a measure of day-to-day variation in measurement results within a test lab. The day-to-day average results for each lab are shown in figure 4. Each laboratory average is indicated by the horizontal bar with error bars corresponding to ± 0.85 vol %. The open symbols indicate the daily average results. The data from labs 1, 4 and 9 had the best repeatability. One implication of poor repeatability is that the results of a single measurement trial may fail to

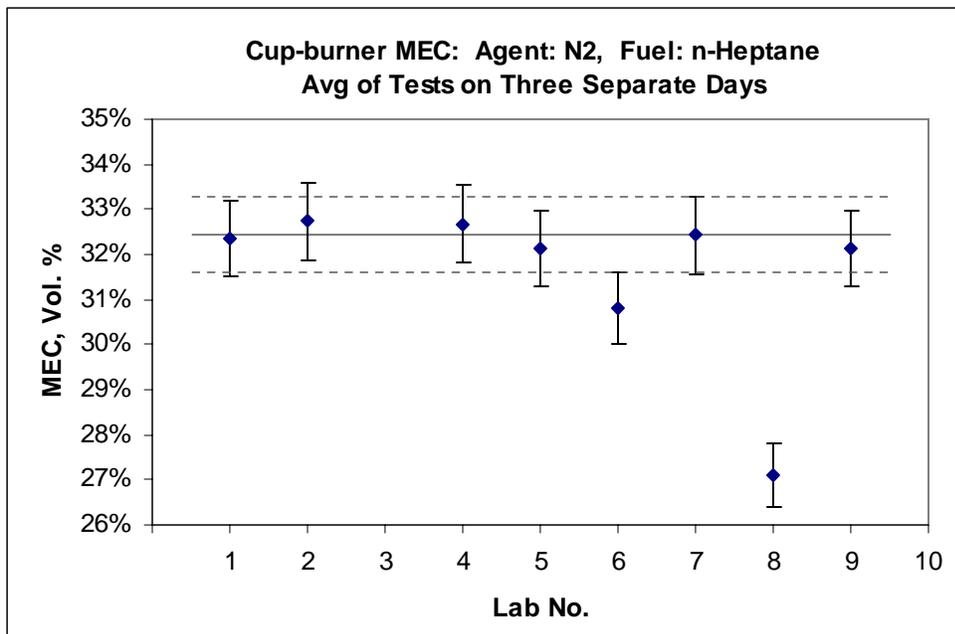


Figure 3. Average laboratory cup-burner nitrogen MEC values based on determinations made on three separate days. The solid lines show the consensus mean (32.44 vol %) and the dotted lines are ± 1 standard deviation (0.85 vol %) from the mean.

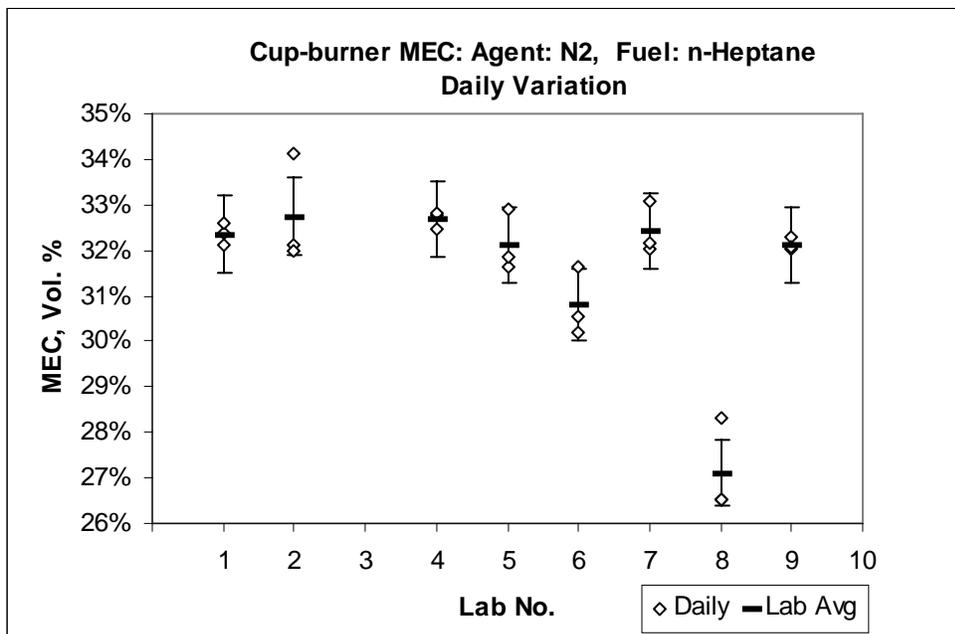


Figure 4. Daily determinations of nitrogen MEC values in relation the laboratory mean and group standard deviation.

accurately represent the MEC of an agent for a particular fuel. Inaccurate MEC values have implications for the actual design safety margin of a fire extinguishing system as well as for the competitive position of the product. For example, if the Lab 2 measured and reported the MEC of a nitrogen-like agent heptane based solely on data represented by the highest open symbol in figure 3, 34.1 vol %, then the manufacturer may be required to market the product at a competitive disadvantage of 5% on agent quantity.

HFC-227ea. Table 2 shows the test result set for the five laboratories that participated in the HFC-227ea extinguishing tests. The daily averages are based on the five extinguishing values reported for the day. The standard deviation value for each lab was calculated based on all 15 reported data points.

Lab #	Day 1	Day 2	Day 3	3-day Average	3-day Range	St Dev*	Range/ St Dev
1	6.61%	6.67%	6.55%	6.61%	0.12%	0.15%	0.78
2	6.58%	6.65%	6.60%	6.61%	0.07%	0.14%	0.51
3	6.74%	6.59%	6.69%	6.67%	0.14%	0.14%	1.06
4	6.59%	6.59%	6.60%	6.59%	0.01%	0.09%	0.11
7	6.32%	6.37%	6.28%	6.32%	0.09%	0.13%	0.70

* Standard deviation based on all 15 reported measurements, five each on three days.

The three-day laboratory average results are shown graphically in figure 5. The results of four labs were in very close agreement and served as the basis of calculating the consensus average MEC for HFC-227ea as 6.62 vol % for n-heptane flames. The dotted lines represent the bounds of the four-lab average plus and minus one standard deviation of 0.13 vol % (2.0% of the consensus mean) which was calculated based on all 75 HFC-227ea data points submitted. These data serve to establish the position that the revised cup-burner test method and apparatus are fully capable of providing highly reproducible results for halocarbon gaseous extinguishing agents.

The average results for Lab 7 was 6.32 vol % which is 2.3 standard deviations below the consensus mean.

Repeatability of these data is shown by the day-to-day average results for each lab shown in figure 6. The average MEC value for each laboratory is indicated by the horizontal line marker with error bars corresponding to ± 0.13 vol %. The open symbols indicate the daily average results. The data from all labs seems to be tightly grouped suggesting good repeatability.

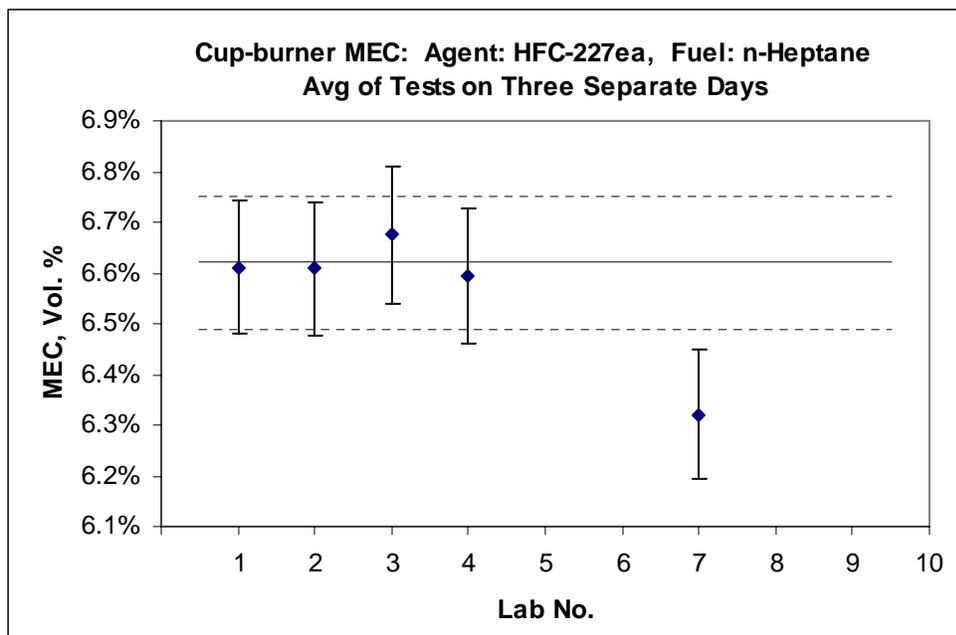


Figure 5. Average laboratory cup-burner HFC-227ea MECs based on determinations made on three separate days. The lines show the mean and ± 1 standard deviation values of the six closely grouped results

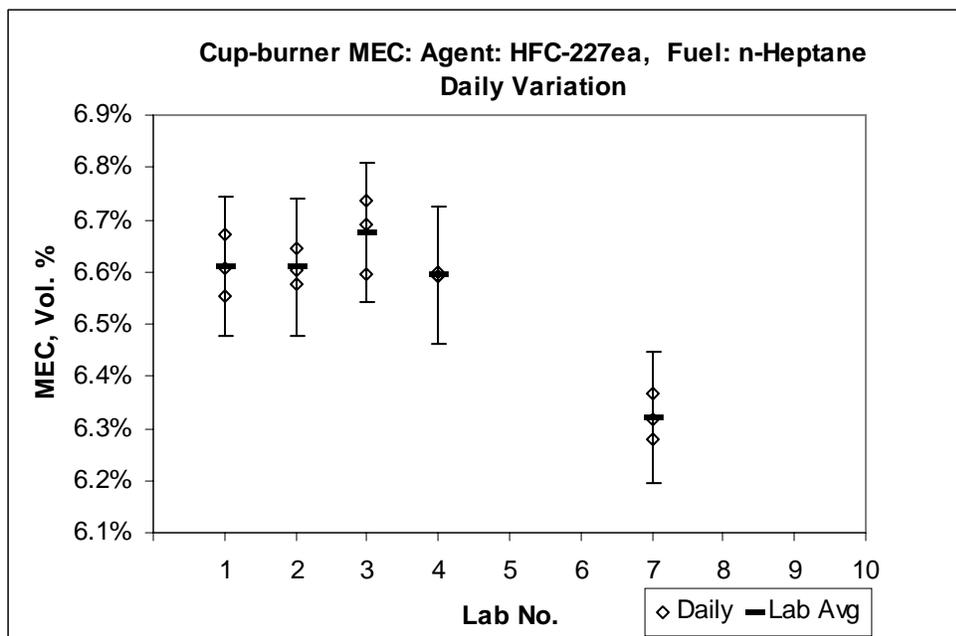


Figure 6. Daily determinations of HFC-227ea MECs in relation to the laboratory mean and group standard deviation.

Application of benchmark data. A question at this point is how to best employ the benchmark values of MEC as an aid to harmonizing extinguishing concentration measurements. The inter-laboratory study data shows that most laboratories using the standard NFPA cup-burner apparatus and procedure produced MEC values in very close agreement. Still, the results from some laboratories differed significantly from the consensus mean values. The consensus mean MEC values (benchmark data) can be used to harmonize data from laboratories so that results, regardless of where determined, are reported on a common basis. Figure 7 contains a graphic that facilitates discussion of an approach to harmonizing MEC data.

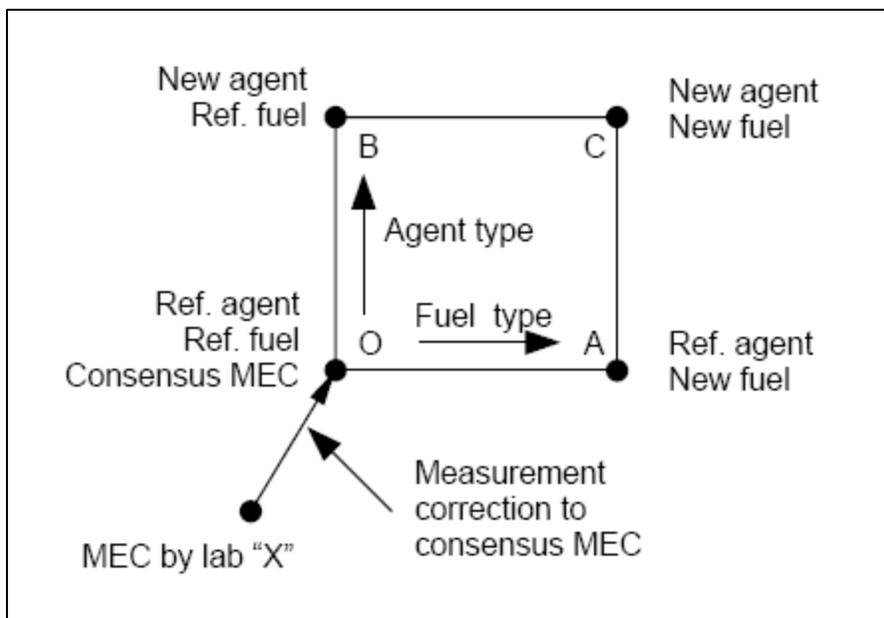


Figure 7. Agent-fuel test domain.

1. Measurement correction factor – MCF. The MEC values of test laboratory “X” may differ from the consensus mean value due to systematic or random errors in procedure, apparatus or test conditions. These are represented by points “X” and “O” in figure 7. If the causes of systematic errors are addressed as well as possible and differences remain between consensus MEC_O and laboratory MEC_X values then a measurement correction factor, MCF, should be calculated as follows:

$$MCF = \frac{MEC_O}{MEC_X}$$

where MEC_O = consensus mean MEC value
 MEC_X = laboratory X’s MEC value

Thus, a laboratory can correct its MEC value to the consensus mean by multiplying by the MFC. A laboratory may have a different MCF value for halocarbon agents and inert gas agents. It is assumed, but not proven that an MCF determined based on testing HFC-227ea

and heptane would apply to MECs determined using other halocarbon agents and fuels. Similarly, it is assumed, but not proven that an MCF determined based on testing nitrogen and heptane would apply to MECs determined using other inert gas agents and fuels.

2. Point “A” in figure 7 represents the case of testing a new fuel, say 1-propanol, with a reference agent (HFC-227ea or nitrogen). A laboratory can obtain a harmonized value of MEC by multiplying the experimentally determined MEC by the MCF. There is some uncertainty on the accuracy of this correction since the cause of the original systematic difference in MEC may have been fuel and flame related.
3. Point “B” in figure 7 represents the case of testing a new agent⁸ with the reference fuel. A laboratory can obtain a harmonized value of MEC by multiplying the experimentally determined MEC by the MCF. Again, the assumption here is that the MCF is the same within an agent class, inert gas or halocarbon. There is some uncertainty on the accuracy of this correction since the cause of the original systematic difference in MEC may have been related to the ability to accurately determine agent concentration in air and the same difficulty may occur with other agents.
4. Point “C” in figure 7 represents the case of testing a new agent and new fuel, say IG-55 and 1-propanol. Again, the MCF should be applied to the as-measured MEC value. There is greater uncertainty in the applicability of MCF as the original source of systematic error may be related to both fuel and agent issues.

Where a measurement correction factor is applicable its value will likely be different for inert gas and halocarbon agents.

The suggested “measurement correction factor” approach is not perfect, particularly inasmuch as it would be used to correct a systematic measurement deviation of unknown cause. If the cause, or causes, of measurement deviation is agent dependent or fuel dependent then the suggested corrections to MEC for fuel-agent combinations different from the reference materials may not be valid. In the absence of a better method, however, the MCF approach offers a means to bring an MEC measurement into close agreement with consensus measurements.

Summary. This paper reports the results of an inter-laboratory to evaluate the suitability of a standardized cup-burner test procedure and apparatus for achieving highly reproducible determinations of flame extinguishing concentrations for gaseous agents and liquid fuels. Nine laboratories participated in the study which evaluated a representative halocarbon agent, HFC-227ea, and inert gas agent, nitrogen, on a single flammable liquid fuel, n-heptane. Six of eight laboratories reported determinations of MEC for nitrogen in close agreement with a consensus value of 32.44 ± 0.85 vol %. Four of five participating laboratories reported determinations of MEC for the HFC-227ea in close agreement with a consensus value of 6.62 ± 0.13 vol %. Examination of day-to-day variations in reported data showed that repeatability of HFC-227ea MEC measurements was very good. The repeatability of nitrogen MEC measurements, while generally good, had somewhat greater range and suggests the importance of making multiple MEC determinations in some cases. The MEC values reported by some laboratories differed by

⁸ For example HFC-125, or FK-5-1-12 in the case of halocarbon agents; or IG-541 or IG-55 in the case of inert gas agents)

more than two standard deviations from the consensus mean MEC benchmark values. One approach to harmonizing MEC data is suggested whereby a measurement correction factor (MCF) be employed, where appropriate, in cases where systematic measurement deviation from the consensus mean MEC can not otherwise be resolved.

References

ISO 14520-Part 1, "Gaseous fire-extinguishing systems - Physical properties and system design," International Organization for Standardization, 2nd ed. (2006).

NFPA 2001, "Standard for Clean Agent Fire Extinguishing Systems," National Fire Protection Association, Quincy, MA (2004).

Senecal, Joseph A., "Flame extinguishing in the cup-burner by inert gases," *Fire Safety Journal*, Volume 40, Issue 6, pp. 579-591 (September 2005).

Senecal, Joseph A., "Revised NFPA Cup-burner Test Method: Improving Reproducibility," Halon Options Technical Working Conference, Albuquerque, NM (May 14-16, 2006).

UL-2127, "Inert Gas Clean Agent Extinguishing System Units," Underwriters Laboratories Inc. (UL), 333 Pfingsten Road, Northbrook, IL 60062-2096 (June 30, 2001).

UL-2166, "Halocarbon Clean Agent Extinguishing System Units," Underwriters Laboratories Inc. (UL), 333 Pfingsten Road, Northbrook, IL 60062-2096 (March 22, 2001).