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Microscale Combustion Calorimeter: Interlaboratory Study of Precision and Bias

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16. Abstract

The microscale combustion calorimeter (MCC) was developed by researchers at the Federal Aviation Administration (FAA) as a tool to evaluate research quantities of new materials. The MCC was licensed by the FAA for manufacture. Since then, many have been made and sold around the world. The FAA performed an interlaboratory study under the guidance of the American Society for Testing and Materials (ASTM) to evaluate the precision and bias of the MCC test method, ASTM D7309. This study encompassed MCCs made by several vendors and run by operators in different laboratories. Identical sets of five polymeric materials were sent to the laboratories for evaluation in the MCC. The laboratories were asked to test the samples in triplicate and report the values obtained for heat release capacity, peak heat release rate, total heat release, peak heat release temperature, and residual mass. Over 20 laboratories were asked to participate in the study. Twelve of these laboratories were able to provide data. Statistical analysis was performed on the results from the different laboratories for comparison to each other. The repeatability and reproducibility of the equipment and method were examined. Additional tests were run in the fire science laboratory at the FAA using thermogravimetric analysis to calculate the properties that are measured in the MCC. These tests were used to validate the results obtained by MCC using an alternate technique. Results from the study were very good compared to other fire tests, with a repeatability of <1% to 3.8% and a reproducibility of 2.2% to 7.9%. Recommendations for modifying the MCC methodologies were formulated and are discussed in this report to improve the results for future studies.

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LIST OF SYMBOLS AND ACRONYMS

β	Heating rate
h_c^0	Net heat of combustion
$h_{c,v}$	Heat of combustion of sample gases
m	Sample mass
M'	Specific mass loss rate
η_c	Heat release capacity
N_2	Nitrogen
O_2	Oxygen
P	Thermal combustion property
Q'	Heat release rate
Q_∞,	Total heat release
σ	Standard deviation
T	Temperature
T_{onset}	Onset temperature of decomposition
T_p	Peak heat release temperature
μ	Char residue
ASTM	American Society for Testing and Materials
cm	Centimeters
COV	Coefficient of variation
FAA	Federal Aviation Administration
g	Grams
HIPS	High-impact polystyrene
ILS	Interlaboratory Study
J	Joules
K	Degrees Kelvin
MCC	Microscale combustion calorimeter
min	Minutes
mg	Milligrams
PMMA	Polymethylmethacrylate
PP	Polypropylene
PC	Polycarbonate
PPSU	Polyphenylsulfone
PCFC	Pyrolysis combustion flow calorimetry
s	Seconds
SDTA	Simultaneous differential thermal analyzer
TGA	Thermogravimetric analyses
W	Watts

EXECUTIVE SUMMARY

The microscale combustion calorimeter (MCC) was developed by researchers at the Federal Aviation Administration (FAA) as a tool to evaluate research quantities of new materials. The MCC was licensed by the FAA for manufacture. Since then, dozens have been made and sold around the world. An interlaboratory study has been performed by the FAA under the guidance of the American Society for Testing and Materials (ASTM) to evaluate the precision and bias of the MCC test method, ASTM D7309. This study encompasses MCCs made by several vendors and run in different laboratories by different operators. Identical sets of five polymeric materials were sent to the laboratories for evaluation in the MCC. The laboratories were asked to test the samples in triplicate and report the values obtained for heat release capacity, peak heat release rate, total heat release, peak heat release temperature, and residual mass. Over 20 laboratories participated in the study, of which 12 were able to provide data. Statistical analysis was performed on the results from the different laboratories for comparison. The repeatability and reproducibility of the equipment and method were examined and are discussed in this report. Additional tests were performed in the fire science laboratory at the FAA using thermogravimetric analysis to calculate the properties measured in the MCC. These tests were used to validate the results obtained by MCC using an alternate technique. When compared to other fire tests, the results from the study were better with a repeatability for individual laboratories of <1% to 3.8% and a reproducibility between laboratories of 2.2% to 7.9%. Recommendations for modifying the MCC methodologies were formulated and discussed to improve the results for future studies.

1. INTRODUCTION.

The microscale combustion calorimeter (MCC) was first introduced by the Federal Aviation Administration (FAA) in 1996 [1] and developed into a quantitative measurement technique over the next several years [2-4]. The MCC has existed in its present form since 2004 when it was licensed for manufacture and sale by the FAA. The MCC was developed by researchers at the FAA to evaluate the flammability of milligram-sized research samples of new polymeric materials being developed in the Fire Resistant Materials Program [5]. The MCC combines principles of analytical pyrolysis, gas phase combustion, and oxygen consumption calorimetry into a quantitative method called pyrolysis combustion flow calorimetry (PCFC). The nonflaming combustion test takes 10 to 15 minutes to complete and measures heat release rate Q'(W/g) and temperature T(K) versus time t(s), from which thermal combustion properties are obtained that correlate with bench- and large-scale fire performance. Thermal combustion properties, obtained directly from the heat release rate history $Q'(t/T)$ measured during the test, include the maximum (peak) heat release rate Q'_{max}, the temperature T_p at Q'_{max}, the total heat of combustion of the sample gases Q_∞, the temperature at the onset of thermal decomposition/heat release T_{onset}, and the mass fraction of the sample remaining after the test (pyrolysis residue or char yield). A derived quantity called the heat release capacity, $\eta_c = Q'_{max}/\beta$, that accounts for the effect of heating rate on Q'_{max} is also obtained. In 2007, PCFC was adopted by the Thermal Properties of Plastics Subcommittee (D20.30) of the American Society for Testing and Materials (ASTM) [6] as a standard method for determining flammability characteristics of plastics and other solid materials. The MCC is one embodiment of that standard, as dozens of MCCs conforming to ASTM D7309 are in use around the world.

A precision statement must accompany the ASTM D7309 standard. Precision is the closeness of agreement between independent test results obtained under a stipulated procedure, in this case ASTM D7309-07. Precision has two components: repeatability and reproducibility. Repeatability is the closeness of agreement between independent measurements obtained by the same method on identical specimens in the same laboratory by the same operator using the same equipment in a short period of time, i.e., it is a measure of the variation within a laboratory. Reproducibility is the closeness of independent measurements obtained by the same method on identical specimens in different laboratories by different operators using different equipment, i.e. a measure of the variation between laboratories. Accuracy is the closeness of a measured quantity to its true value. Bias is a systematic deviation of the measured value from the true value.

This report documents the results of an interlaboratory study (ILS) conducted by the FAA under the auspices of ASTM according to their standard practice [7] for producing the required precision statement. Over 20 different laboratories were solicited for the ILS. Twelve of these laboratories used MCC equipment obtained from three different manufacturers constructed according to ASTM D7309. The precision results from the 12 laboratories are presented along with a basic statistical analysis of these data. The accuracy of ASTM D7309, while not part of the ILS, was obtained by comparing the ILS results from the FAA laboratory for the thermal combustion properties of the five polymer samples to the thermal combustion properties of the same samples measured by a different method of thermal analysis (thermogravimetry).

2. EXPERIMENTAL.

2.1 MATERIALS.

Samples of five different polymers were sent to each of the 12 participating laboratories. The polymers, which are listed in table 1, are polymethylmethacrylate (PMMA), high-impact polystyrene (HIPS), polypropylene (PP), bisphenol-A polycarbonate (PC), and polyphenylsulfone (PPSU). These samples were selected to provide a wide range of thermal combustion properties in the MCC. The properties of interest were the peak heat release rate Q'_{max}, the temperature at Q'_{max}, the total heat of combustion per unit mass of sample Q_∞, the mass fraction of the sample remaining after the test (pyrolysis residue and char yield) μ, and the heat release capacity $\eta_c = Q'_{max}/\beta$, where $\beta = 1$ K/s is the constant heating rate specified for the ILS tests. Figure 1 shows Q' versus temperature data for each of the five polymers. Each polymer has distinct Q'_{max}, Q_∞, T_p, and μ covering a wide range of values.

Table 1. Sample Specifications

Sample	Abbreviation	Specimen Mass (mg)	Supplier
Polymethylmethacrylate	PMMA	3.18	U.S. Plastic Corp.
High-impact polystyrene	HIPS	4.55	U.S. Plastic Corp.
Polypropylene	PP	3.44	U.S. Plastic Corp.
Poloycarbonate	PC	3.47	U.S. Plastic Corp.
Polyphenylsulfone	PPSU	6.20	Evonik Industries

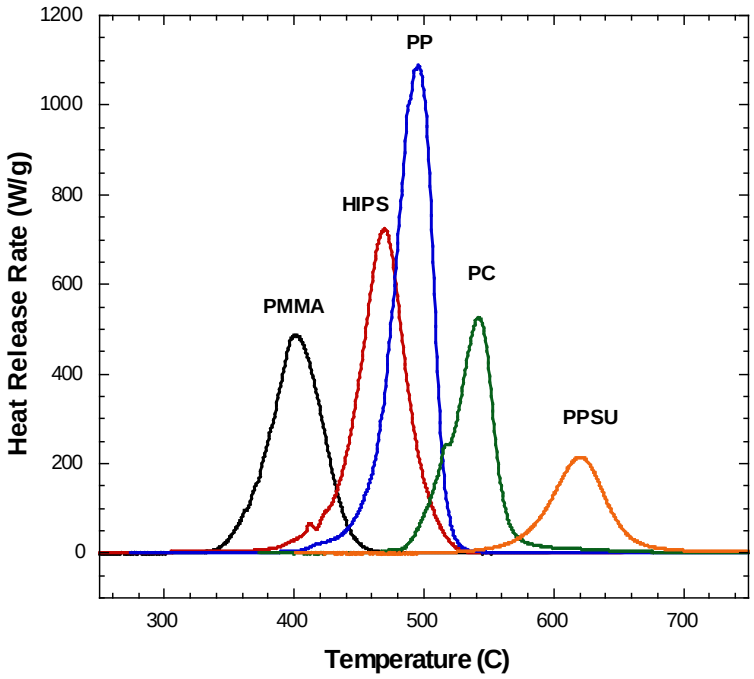

Figure 1. Heat Release Rate Q' vs Temperature Data According to ASTM D7309-07 for the Five Polymers in the ILS

Samples of each polymer of consistent sizes and weights were provided to the participating laboratories in the form of cylindrical pellets that were hole punched from thin films. Punched samples and films of different diameters were used to obtain sample weights appropriate to the ASTM D7309 test protocol in an effort to eliminate sample handling and preparation as a source of error. The sample weights ranged from 3.2 to 6.2 mg. Table 1 shows the supplier and average weight of the samples. Supply gases used by the participants were of unknown purity. However, ASTM D7309 specifies that the purity of the gases be greater than 99.5%.

2.2 METHODS.

2.2.1 The MCC.

The five polymer samples were tested in the MCC as received. Each laboratory ran triplicate determinations according to ASTM D7309-07 Method A [6] in which samples are heated in nitrogen at a rate of β = 1 K/s until decomposition is complete or a temperature of 1173 K (900°C) is reached. The volatile thermal decomposition products are purged from the pyrolyzer, mixed with excess oxygen, and completely oxidized in the combustion chamber. The heat release rate of the sample gases in watts (W) is calculated from oxygen depletion [8-10], and the specific heat release rate Q' in W/g is obtained by dividing the heat release rate by the initial sample mass. The maximum specific heat release rate Q'_{max} and the temperature at maximum heat release rate T_p are two of the five thermal combustion properties measured in the ILS. The heat release capacity $\eta_c = Q'_{max}/\beta$ is a derived quantity [4] that accounts for the effect of heating rate on Q'_{max}. The total heat released by combustion of the pyrolysis gases per unit initial mass

of sample Q_∞ is obtained by numerical integration of Q' versus time or Q'/β versus temperature. The software provided with the MCC is used to obtain these results from the measured Q' versus time or Q'/β versus temperature data. The residual mass fraction or char yield, μ, is obtained by weighing the sample before and after the test. The average heat of combustion of the volatile sample gases for the test is $h_{c,v} = Q_\infty/(1-\mu)$. Triplicate measurements of the five thermal combustion properties, Q'_{max}, T_p, η_c, Q_∞, and μ, were made for each polymer sample by each laboratory, and the data were collected for the ASTM D7309 precision analysis.

In the FAA laboratory, the flow rates and the oxygen sensor were calibrated according to ASTM D7309-07, and the temperature at the sample location was calibrated according to a standard method [11] using the differential temperature signal at the sample position to locate the onset of melting of pure metals in standard alumina sample cups. Tests were also conducted according to ASTM D7309, Method B, using an air purge gas to measure the total heat of combustion of the polymer (gases + char).

2.2.2 Thermogravimetric Analysis.

Thermogravimetric analyses (TGA) were performed on the five polymer samples of the ILS in the FAA laboratory using a commercial instrument (Mettler-Toldedo SDTA 851e) according to a standard method [12]. Tests in the TGA were conducted under a N_2 purge at 80 cm^3/min and a heating rate of 1 K/s. Alumina sample cups from the MCC were used in the TGA to reproduce conditions in the MCC as closely as possible. The samples were tested until thermal decomposition was complete or until 900°C was reached. Temperature calibration was performed according to the same standard method [11] used for the MCC in which using the onset of melting of pure metal standards at 1 K/s (60 K/min) in the standard alumina MCC sample cups.

3. RESULTS.

3.1 PRECISION (REPEATABILITY AND REPRODUCIBILITY).

The results from the ILS study were encouraging with regard to the precision of the results obtained using ASTM D 7309-07. After the data were collected it was discovered that many of the participating laboratories did not analyze and interpret the data correctly. The major sources of variation were inconsistent baseline correction, which effected Q'_{max} and Q_∞, and the choice of the maximum in the Q' history to use in the η_c calculation [13]. This component of the interlaboratory variation due to operator error was eliminated by collecting and reanalyzing all the raw data from the 12 laboratories at the FAA using the same operator, software, and protocol. Thermal combustion properties T_p and μ were not significantly affected by the baseline and Q' history interpretations.

The complete results of the ILS are given in the appendices. Repeatability and reproducibility are plotted in figures 2 through 6 as the standard deviations of the data within each laboratory (repeatability standard deviation) and between all of the laboratories (reproducibility standard deviation) against the global average of each thermal combustion property P for each material

obtained from all the laboratories. An in-depth statistical analysis is available from ASTM ILS Report 589 [14].

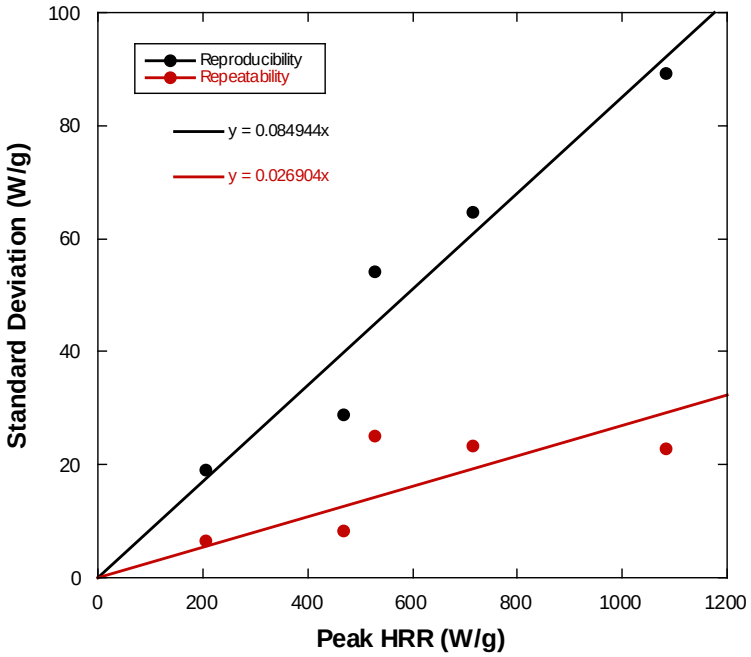

Figure 2. Repeatability and Reproducibility of the Peak Heat Release Rate

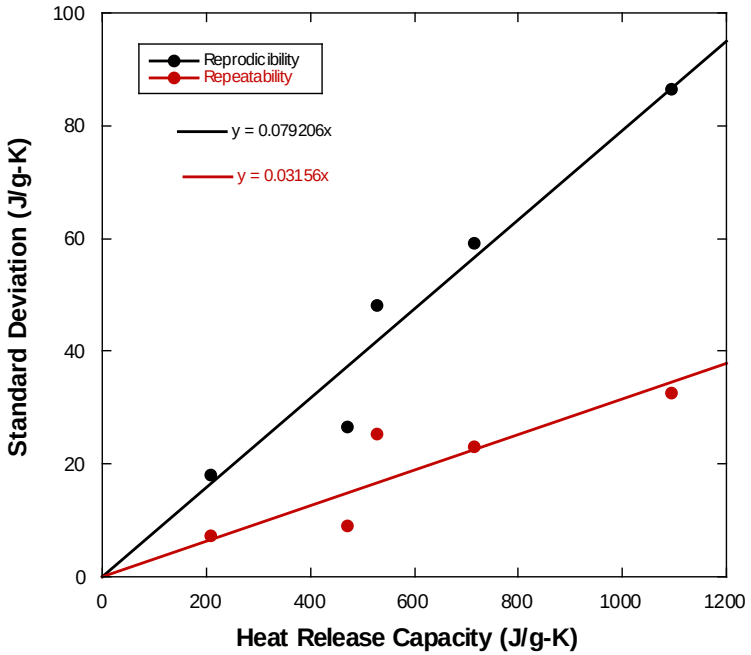

Figure 3. Repeatability and Reproducibility of the Heat Release Capacity

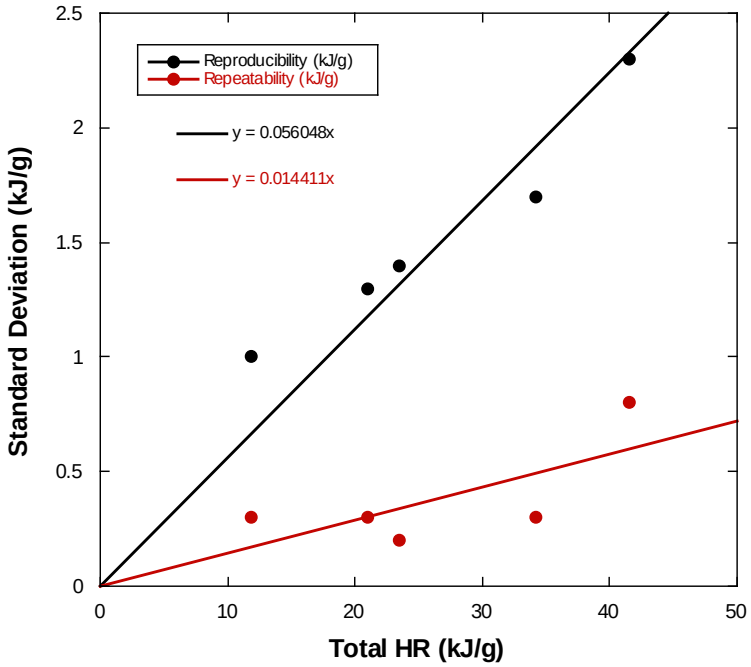

Figure 4. Repeatability and Reproducibility of the Total Heat Release Q_{∞}

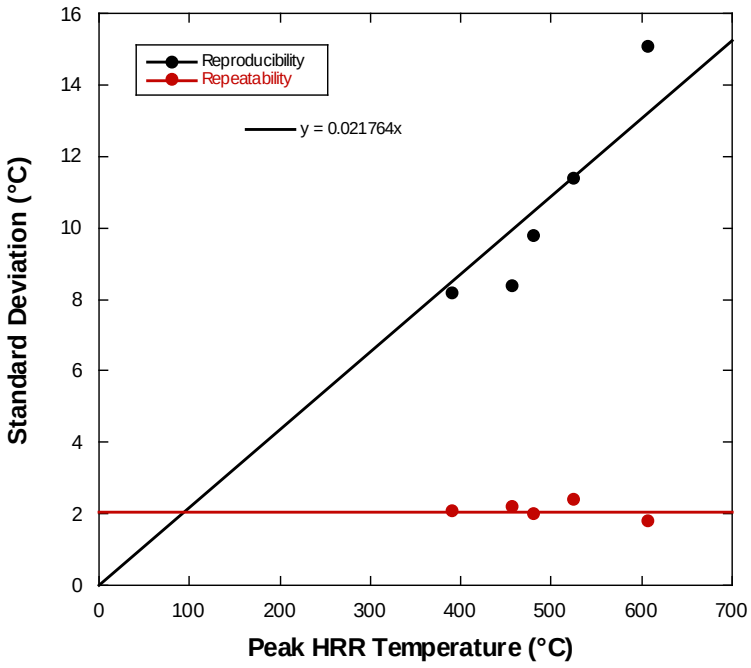

Figure 5. Repeatability and Reproducibility of the Temperature T_p at $Q'_{\max}$

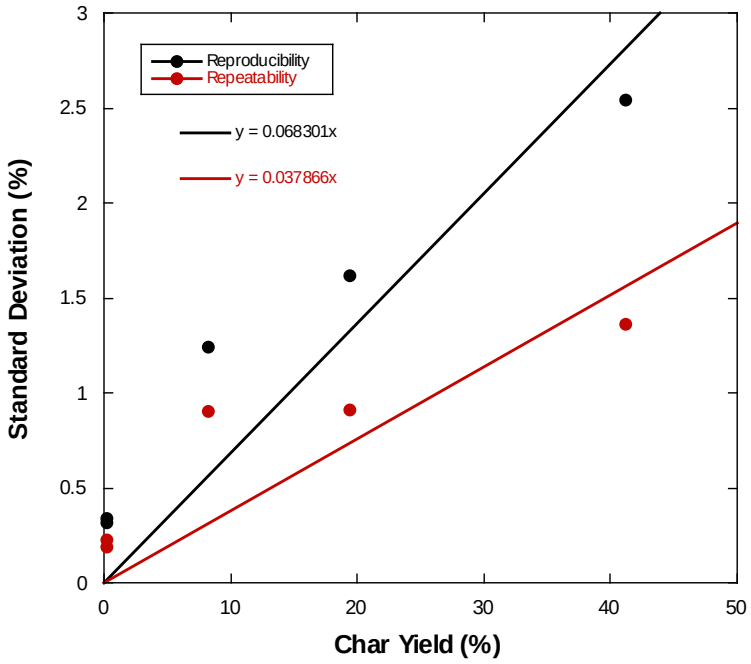

Figure 6. Repeatability and Reproducibility of the Char Yield μ

It is immediately obvious from figures 2 through 6 that the standard deviations, σ, of the thermal combustion properties repeatability and reproducibility are roughly proportional to the value of the thermal combustion property P (with the exception of the repeatability of T_p), i.e., σ/P = constant = average coefficient of variation for all P (polymers). The average coefficient of variation (COV) for each property over all of the polymers can be estimated from the slopes of the data plotted in figures 2 through 6, i.e., average COV (%) = slope of σ versus P multiplied by 100. The COVs for repeatability and reproducibility for each thermal combustion property were averaged over all the polymers (table 2).

Table 2. Average COV for Repeatability and Reproducibility of Thermal Combustion Properties for the Polymers in the ILS

Thermal Combustion Property	Repeatability COV (%)	Reproducibility COV (%)
Q'_{max}	2.7	5
η_c	3.2	7.9
Q_∞	1.4	5.6
T_p	<1	2.2
μ	3.8	6.8

3.2 ACCURACY (COMPARISON OF THERMAL COMBUSTION PROPERTIES FROM TGA AND MCC).

Figure 7 shows the mass fraction versus sample temperature data obtained by TGA for each polymer of the ILS. Three of the materials left a thermally stable pyrolysis residue consisting of char (PPSU and PC) or mineral filler (HIPS). Table 3 lists the thermal properties and their values obtained from the TGA tests that directly compare to the MCC tests; these include the onset temperature of decomposition T_{onset} (not reported in the ILS), the maximum mass loss rate M_{max}, the temperature at the maximum mass loss rate T_p, and the char fraction μ.

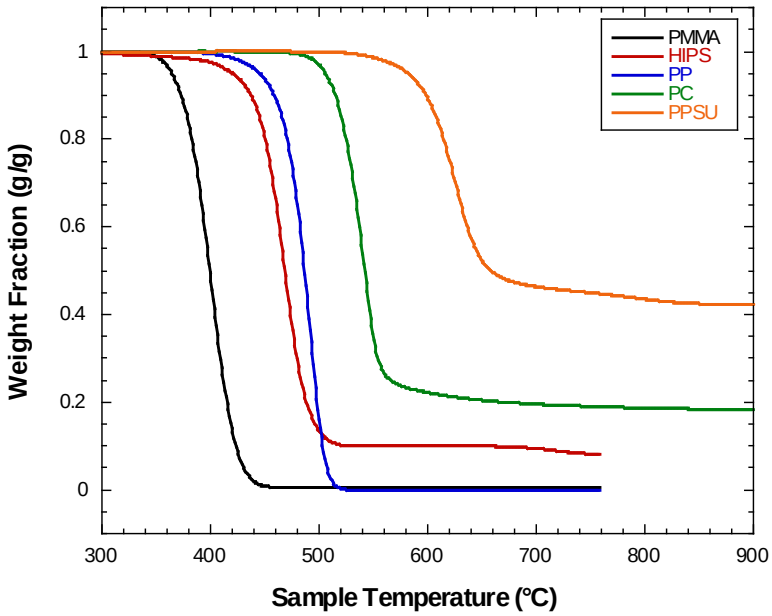

Figure 7. Thermogravimetric Analyses of the Five Polymers Examined in This Study at Heating Rate β = 1 K/s

Table 3. Thermogravimetry Data (Averages of Three Measurements)

Sample	β (K/s)	T_{onset} (°C)	T_p (°C)	M'_{max} (s^{-1})	μ (%)
PMMA	1.009	376	399	0.0204	0.7
HIPS	1.011	444	467	0.0186	8.3
PP	1.005	468	489	0.0264	0.2
PC	1.010	516	540	0.0213	18.3
PPSU	1.010	594	622	0.0104	42.0

The specific mass loss rate in the TGA, M'(g/g-s), is the time derivative of the data in figure 7

$$M'(t) = \frac{-\beta}{m_0}\frac{dm}{dT} = \frac{-1}{m_0}\frac{dm}{dt} \tag{1}$$

In equation 1, m is the instantaneous sample mass, dm/dt is the time derivative (mass loss rate), and m_0 is the initial weight of the sample. The maximum specific mass loss rate due to thermal decomposition during a temperature scanning experiment at heating rate β is $M'_{\max}$. Multiplying equation 1 by the heat of combustion of the sample gases, $h_{c,v}$, gives the specific heat release rate measured in ASTM D7309-07

$$Q'_{TGA} = -\frac{h_{c,v}}{m_0}\frac{dm}{dt} = h_{c,v}M' \tag{2}$$

The heat of combustion of the sample gases, $\Delta h_{c,v}$ (J/g-gas), can be calculated from the data obtained in the MCC

$$\Delta h_{c,v} = \frac{Q_\infty}{1\mu} \tag{3}$$

The heat release capacity as measured in the TGA is calculated by dividing equation 2 by the average heating rate in the test β at the maximum rate mass loss rate

$$\eta_{TGA} = \frac{\Delta h_{c,v}}{\beta}M'_{\max} \tag{4}$$

The results of these calculations are shown in table 4 as average values from the triplicate analysis performed in the FAA laboratory. For comparison to $h_{c,v}$, the net heat of combustion of the entire sample (gases + char) was determined by oxygen bomb calorimetry [10 and 15-17] and ASTM D7309 Method B and is listed in table 4 as h_c^0. These data confirmed the expected result that, for noncharring materials PMMA and PP, the average heat of combustion of the pyrolysis gases determined in the MCC equals the heat of combustion of the polymer ($h_{c,v} = h_c^0$). For the charring materials, PC and PPSU, the heat of combustion of the volatiles is less than the heat of combustion of the polymer ($h_{c,v} < h_c^0$), because the carbonaceous char that remains after the test has high-combustion value. The TGA heat release rate histories Q'_{TGA} were calculated from the instantaneous mass loss per equation 2 using the $h_{c,v}$ in table 4. The results for the five polymers are shown in figure 8, which shows that the Q'_{TGA} and Q' histories superimpose. The superposition of Q' data from the MCC and TGA confirmed the high fidelity of the MCC temporal resolution and suggested the TGA is a standard, independent reference measurement suitable for a determination of MCC accuracy.

Table 4. Net Heat of Combustion of Polymers (h_c^0) and Their Volatile Thermal Decomposition Products ($h_{c,v}$) From ASTM D4809 and ASTM D7309

	ASTM D4809 and D7309 (B)	ASTM D7309 (A)		
Sample	h_c^0 (kJ/g-sample)	Q_∞ (kJ/g)	μ (%)	$h_{c,v}$ (kJ/g-gas)
PMMA	24.9 - 25.1	24.9	0.7	25.1
HIPS	39.7 - 42.5	35.4	8.3	38.6
PP	42.7 - 43.2	42.5	0.2	42.6
PC	29.8 - 30.4	22.0	18.3	26.9
PPSU	27.2 - 29.2	13.1	42.0	22.6

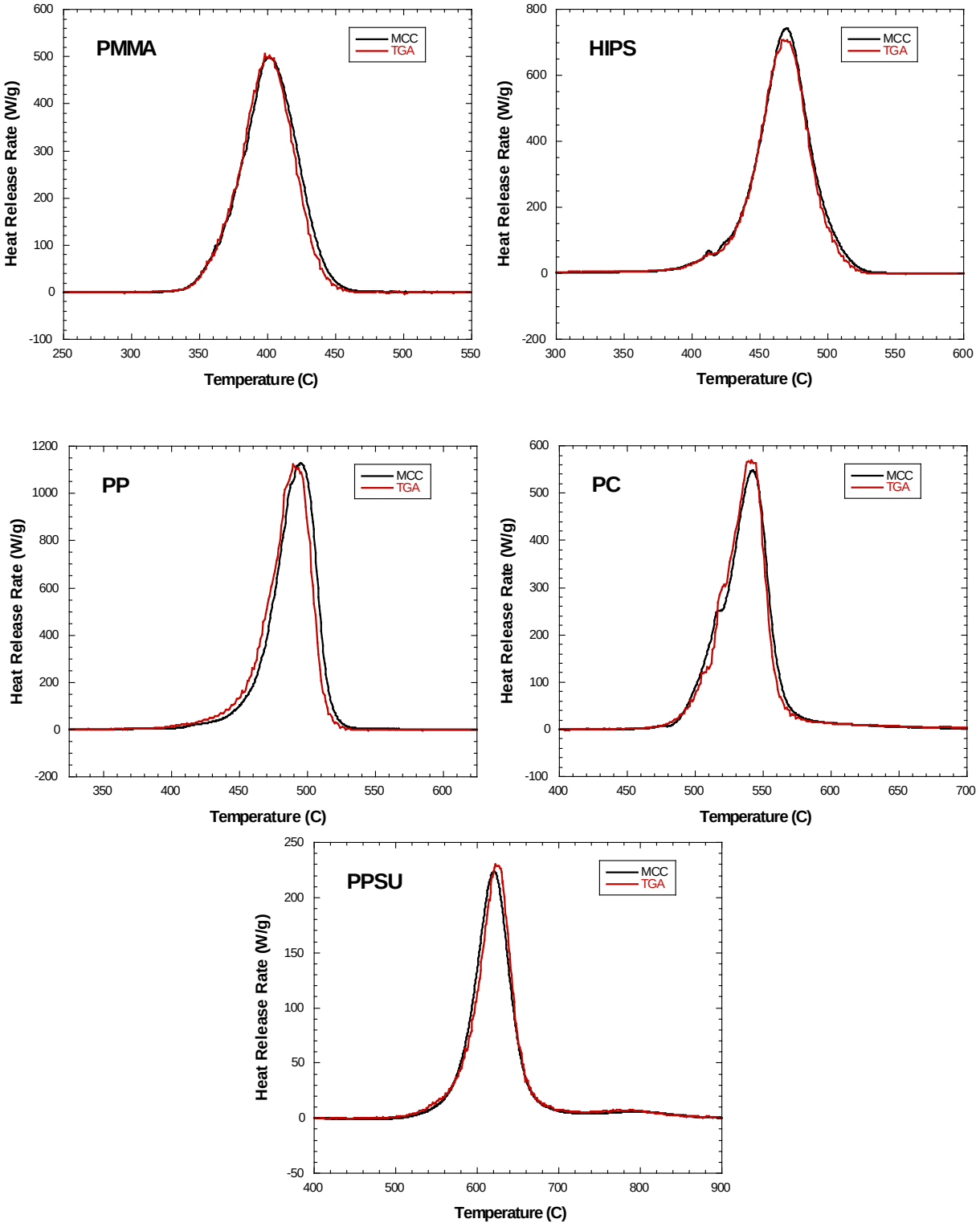

Figure 8. Comparison of Q' Measured in the MCC to Q'_{TGA} Calculated From TGA Data and Equation 2 for the Polymers of This Study

The thermal combustion properties μ, T_p, and η_c measured by TGA and MCC are listed in table 5 along with the standard deviations calculated from the triplicate measurements.

Table 5. Comparison of Thermal Combustion Properties From TGA, MCC per ASTM D7309 at β = 1K/s

	Char Yield, μ (%)		Decomposition Temperature, T_p (K)		Heat Release Capacity, η_c (J/g-K)	
Sample	TGA	ASTM D7309	TGA	ASTM D7309	TGA	ASTM D7309
PMMA	0.7 ±0.1	0	672 ±2	675 ±1	508 ±4	495 ±5
HIPS	8.3 ±0.1	8.8 ±0.1	740 ±2	742 ±1	710 ±50	752 ±8
PP	0.2 ±0.3	0	762 ±2	768 ±1	1116 ±17	1149 ±90
PC	18.3 ±0.0	18.7 ±0.3	813 ±2	814 ±2	569 ±4	546 ±17
PPSU	42.0 ±0.1	40.4 ±0.0	895 ±0	895 ±1	232 ±2	227 ±2

4. DISCUSSION.

There are several sources of error in the heat release measurements of ASTM D7309-07. First is the data acquisition board, which when out of calibration, can have small effects on the flow meter and flow controllers, but large effects on the temperature and oxygen concentration measurements.

Another source of error is the sample weight measurement. A propagation of error analyses determined that the sample weighing is the largest source of error of the measurements used to calculate the heat release rate. Uncertainty in the measurement and repeatability issues can affect the results considerably. Balances used to weigh samples for the MCC should be accurate to a minimum of 0.01 mg. Also, sample mass should be sufficient to provide a good signal-to-noise ratio at half-scale deflection of the oxygen concentration. Samples provided to the ILS participants were of appropriate mass and should have been tested as-received. Participants were asked to record the sample weights for statistical analysis and as a check to ensure the balances were operating properly.

Faulty flow rate calibrations and leaks in the system also contributed to the measured heat release value errors. Several laboratories conducted tests without first determining that the total flow rate was within specification (100 ±1 cm^3/min). If the total flow measured at the instrument outlet is not equal to the sum of the purge gas (N_2) and oxidizer (O_2) flows, there is a system leak, or the flow meters are out of calibration. Leaks can occur at the sample platform flange and the scrubber tubes, which can usually be fixed by greasing the O-ring seal of the sample flange and cleaning and reseating the fittings for the scrubber tubes. Less common leak points are the ceramic-to-metal junctions of the combustion tube that can appear after a large number of tests (thermal cycles). These can be fixed by tightening the fittings or, if necessary, replacing the O-rings.

A temperature calibration procedure [11] was adopted recently in the FAA laboratory that is in general use for thermal analysis equipment, but is not specified in ASTM D7309-07. This calibration helps correct thermal lag of sample thermocouple during transient heating of the sample. The known melting temperature of high-purity metal standards is compared to the onset of the sample deviation (knee) and program temperatures in the pyrolyzer, similar to the procedure used for differential scanning calorimeters [18]. The known melting temperatures of the metals are plotted versus the measured values from the MCC to generate a correction factor. A temperature calibration eliminates bias that affects reproducibility but not repeatability (see figure 5).

5. CONCLUSIONS.

This study shows that ASTM D7309-07 generates thermal combustion properties with precision and potentially good accuracy when proper calibrations are performed and appropriate protocols are used to analyze the data. This was the first interlaboratory study (ILS) for the MCC described in ASTM D7309. The manufacturers of the MCC have been consulted on the appropriate calibration schedule of the equipment as well as some engineering improvements they should implement. In addition, a new temperature calibration routine was introduced that should also be adopted by the manufacturers and included in the next revision of the standard. Even without these improvements, the reproducibility and the repeatability were excellent, especially when compared to other fire tests.

More detailed and specific instructions on how to conduct the tests for the ILS should have been given to the operators. In particular, the maximum test temperature should have been stated to ensure the complete decomposition of the sample and help make the char yields more consistent, the samples should have been tested as-received, and a heating rate of β = K/s should have been specified. In the cases where samples were not tested under these conditions, the participants were asked to retest the samples for inclusion in the study.

Sources of error were identified in the sampling, test methodologies, and data interpretation. Also, several design discrepancies were identified and efforts are being made by the manufacturers to correct them. Hopefully, efforts by the manufacturers to educate their customers and implement design and procedure changes that correct these sources of error will improve the reproducibility of ASTM D7309. The FAA is in the process of writing a comprehensive manual on the theory, operation, and calibration of the MCC, as well as data interpretation.

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APPENDIX A—LABORATORY TEST RESULTS

Table A-1. Maximum Heat Release Rates Q'_{max} (W/g)

Laboratory	PMMA	PP	HIPS	PC	PPSU
1	508.5	1203.2	793.6	605.5	239.6
	504.6	1196.4	766.6	606.9	236.3
	506.4	1202.3	800.8	594.6	235.5
2	471.0	1083.0	701.0	502.0	188.0
	463.0	1073.0	721.0	509.0	193.0
	465.0	1098.0	708.0	552.0	199.0
3	486.0	1126.0	748.0	572.0	229.0
	492.0	1128.0	759.0	572.0	222.0
	488.0	1124.0	747.0	583.0	221.0
4	489.9	1110.0	708.9	537.5	213.3
	492.3	1129.0	776.0	507.4	220.1
	494.6	1128.0	763.8	543.5	221.0
5	505.0	1035.0	721.9	614.0	223.0
	509.0	1005.0	787.5	576.0	199.0
	487.5	1003.0	778.4	577.0	189.0
6	478.0	1115.0	730.0	543.0	215.0
	486.0	1038.0	724.0	509.0	217.0
	487.0	1089.0	745.0	526.0	215.0
7	427.3	1166.0	722.3	588.0	186.4
	429.0	1152.0	707.9	547.0	180.1
	434.0	1130.0	687.5	573.0	184.8
8	483.0	1166.0	723.0	527.0	202.0
	454.0	1166.0	706.9	518.0	212.0
	464.0	1122.0	746.0	478.0	201.0
9	456.0	1090.0	748.0	532.0	208.0
	469.0	1097.0	717.0	501.0	209.0
	469.0	1092.0	697.0	485.0	218.0
10	475.0	1146.0	712.0	470.0	NA
	468.0	1102.0	775.0	548.0	NA
	468.0	1144.0	759.0	444.0	NA
11	406.9	920.7	574.7	436.9	186.4
	430.2	924.6	616.0	417.7	179.7
	432.9	876.8	614.5	442.6	188.6
12	436.0	900.0	608.0	447.3	179.4
	438.0	985.0	567.0	502.2	179.4
	417.0	943.0	573.0	481.6	181.5

PMMA = Polymethylmethacrylate
PP = Polypropylene
HIPS = High-impact polystyrene
PC = Polycarbonate
PPSU = Polyphenylsulfone

Table A-2. Heat Release Capacities η_c (J/g-K)

Laboratory	PMMA	PP	HIPS	PC	PPSU
1	498	1161	756	574	239
	491	1161	734	576	231
	493	1167	763	571	235
2	468	1087	690	501	189
	459	1078	714	510	197
	461	1097	697	552	201
3	491	1134	749	574	228
	495	1134	761	574	221
	494	1130	749	585	221
4	490	1111	708	538	213
	491	1130	774	508	220
	495	1127	765	544	221
5	506	1041	724	617	225
	509	999	791	579	202
	490	1012	781	580	192
6	489	1249	750	563	227
	497	1073	745	529	228
	498	1126	761	547	225
7	428	1180	721	587	190
	429	1162	704	547	186
	437	1164	689	573	191
8	483	1173	714	528	202
	446	1172	690	519	213
	465	1126	730	478	203
9	464	1113	761	540	214
	477	1116	727	512	215
	477	1115	716	494	223
10	476	1161	710	472	NA
	473	1117	773	552	NA
	472	1159	759	447	NA
11	426	957	603	454	195
	450	964	642	435	190
	454	920	641	471	214
12	434	896	607	447	179
	436	982	565	502	179
	415	938	576	482	181

PMMA = Polymethylmethacrylate
PP = Polypropylene
HIPS = High-impact polystyrene
PC = Polycarbonate
PPSU = Polyphenylsulfone

Table A-3. Total Heat Release Q_∞ (kJ/g)

Laboratory	PMMA	PP	HIPS	PC	PPSU
1	24.5	42.6	35.4	21.3	13.0
	24.2	42.4	35.1	21.6	13.4
	24.5	42.5	35.2	21.5	13.0
2	22.0	37.8	31.6	20.2	11.5
	22.0	37.8	32.2	20.1	11.9
	22.1	38.5	32.6	19.9	12.2
3	25.1	43.5	36.4	22.4	12.3
	25.1	43.9	36.0	22.4	12.2
	25.2	43.8	36.6	22.3	12.0
4	23.4	42.0	34.3	20.9	11.2
	23.8	42.4	34.5	21.0	11.3
	23.9	42.1	34.2	21.3	11.3
5	25.1	47.8	35.5	22.7	13.3
	25.1	43.6	35.8	22.5	12.7
	25.2	43.8	34.9	22.3	12.5
6	24.9	42.5	35.7	22.1	13.1
	24.9	42.5	35.6	21.8	13.2
	24.9	42.6	35.0	22.0	13.1
7	22.9	41.4	33.1	20.3	11.8
	22.9	42.0	33.9	20.9	12.0
	22.8	41.9	33.6	20.7	12.0
8	22.6	41.5	33.6	20.3	10.8
	22.6	41.3	34.0	20.0	11.7
	22.6	41.2	33.7	20.1	11.3
9	24.5	43.1	35.6	22.5	12.7
	24.6	42.9	35.6	22.5	12.7
	24.6	42.8	35.7	22.9	12.7
10	24.3	42.2	34.5	20.8	NA
	23.7	40.8	34.7	22.2	NA
	23.5	40.7	35.4	20.9	NA
11	21.7	39.2	31.1	19.3	10.8
	22.0	39.4	31.3	19.1	10.5
	22.0	39.4	31.2	19.2	10.8
12	21.7	36.6	32.3	18.6	10.6
	21.5	38.4	32.0	18.7	10.1
	20.7	38.5	31.5	19.1	10.3

PMMA = Polymethylmethacrylate
PP = Polypropylene
HIPS = High-impact polystyrene
PC = Polycarbonate
PPSU = Polyphenylsulfone

Table A-4. Temperature at Maximum Heat Release Rate T_p (°C)

Laboratory	PMMA	PP	HIPS	PC	PPSU
1	406.2	496.9	469.3	541.9	626.4
	404.6	495.5	470.4	542.6	627.5
	405.1	497.6	469.7	541.0	629.5
2	381.0	470.0	445.0	512.0	587.0
	382.0	471.0	447.0	509.0	590.0
	380.0	369.0	445.0	513.0	587.0
3	393.0	489.0	459.0	532.0	614.0
	392.0	484.0	458.0	529.0	616.0
	393.0	488.0	460.0	532.0	616.0
4	391.7	488.8	461.2	534.4	617.2
	391.6	488.9	459.8	533.0	612.9
	392.8	485.7	460.1	536.0	613.7
5	380.0	475.0	446.3	515.2	599.3
	382.2	470.0	446.4	516.5	599.3
	381.0	472.0	448.8	516.1	600.6
6	401.5	495.1	467.4	542.6	622.9
	402.7	494.9	470.0	539.6	621.6
	400.8	496.0	469.1	541.9	620.6
7	388.5	472.0	448.8	516.0	576.7
	387.3	467.0	448.8	516.0	580.4
	390.4	472.0	449.0	513.0	579.9
8	397.0	478.5	455.0	524.0	604.0
	397.0	479.0	455.0	526.0	601.0
	394.0	481.0	454.0	532.0	601.0
9	394.0	489.0	461.0	533.0	617.0
	395.0	489.0	462.0	535.0	618.0
	394.0	486.0	461.0	532.0	618.0
10	383.0	473.0	448.0	514.0	NA
	381.0	473.0	446.0	515.0	NA
	383.0	469.0	445.0	511.0	NA
11	385.1	483.0	456.8	523.2	612.8
	380.7	482.6	458.7	528.0	609.2
	391.9	476.9	452.8	521.4	612.8
12	381.0	473.0	460.0	518.4	599.3
	387.0	473.0	452.0	511.6	593.3
	384.0	474.0	448.0	511.6	594.0

PMMA = Polymethylmethacrylate
PP = Polypropylene
HIPS = High-impact polystyrene
PC = Polycarbonate
PPSU = Polyphenylsulfone

Table A-5. Char Yield μ (%)

Laboratory	PMMA	PP	HIPS	PC	PPSU
1	0.1	0.1	8.7	19.9	40.2
	0.0	0.1	9.0	19.6	40.5
	0.1	0.1	8.9	19.6	40.2
2	0.0	0.0	7.0	18.4	40.6
	0.0	0.0	6.9	17.7	39.7
	0.2	0.0	7.6	19.2	37.0
3	0.6	0.6	9.2	18.5	42.3
	0.0	0.3	8.9	18.4	42.4
	0.0	0.0	8.6	19.1	42.0
4	0.0	0.0	8.0	23.0	44.0
	0.0	0.0	9.0	22.0	42.0
	0.0	0.0	8.0	21.0	42.0
5	0.0	0.0	11.3	19.5	33.6
	0.6	0.0	5.4	21.7	39.5
	0.0	0.0	7.8	19.1	39.4
6	0.0	0.0	8.7	18.4	40.4
	0.0	0.0	8.8	19.0	40.4
	0.0	0.0	8.8	18.6	40.4
7	0.6	0.8	8.3	16.7	40.4
	0.7	0.0	8.8	17.7	41.0
	0.0	0.0	8.7	17.1	42.4
8	0.9	0.4	10.1	21.4	44.6
	0.0	0.2	10.0	21.7	45.9
	0.2	0.4	9.5	22.6	47.0
9	0.0	0.9	9.0	19.1	42.3
	0.0	0.9	9.1	20.8	42.0
	0.0	1.1	8.6	19.9	41.6
10	5.8	0.0	7.2	19.3	NA
	0.0	0.0	7.5	17.3	NA
	0.0	0.0	7.2	20.5	NA
11	0.0	0.0	7.7	19.0	39.2
	0.0	0.0	7.6	18.4	39.8
	0.0	0.0	7.2	18.6	39.0
12	0.8	0.3	6.4	19.9	44.3
	1.1	0.9	2.7	17.6	43.1
	0.7	0.6	6.0	17.3	42.1

PMMA = Polymethylmethacrylate
PP = Polypropylene
HIPS = High-impact polystyrene
PC = Polycarbonate
PPSU = Polyphenylsulfone