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Plastics — Use of polyethylene reference specimens (PERS) for monitoring laboratory and outdoor weathering conditions

Plastiques — Utilisation d'éprouvettes de référence en polyéthylène pour l'évaluation des conditions de vieillissement climatique

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Contents

Page

Foreword

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ISO/TR 19032 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

Introduction

The method described in this Technical Report demonstrates the use of polyethylene reference specimens (hereafter called PERS) for monitoring conditions in weathering tests used for plastics. The PERS has double bonds in its molecular structure, which are easily oxidized to produce carbonyl groups. The change in carbonyl index of PERS is produced by the combined effects of ultraviolet (UV) and temperature. Therefore, the carbonyl groups proportionally increase, depending on the received UV and temperature. Based on this relationship, the effect of UV radiation and temperature on PERS can be expressed quantitatively. For laboratory-accelerated exposures, PERS is also sensitive to changes in the chamber air temperature. The effect of moisture was not determined in the study.

Plastics — Use of polyethylene reference specimens (PERS) for monitoring laboratory and outdoor weathering conditions

1 Scope

This Technical Report describes a method that demonstrates the use of polyethylene reference specimens (PERS) for monitoring laboratory and outdoor conditions in weathering tests used for plastics.

2 Background information

Degradation of plastics in an outdoor environment is mainly influenced by the ultraviolet radiation received, environmental temperature, moisture, etc. Especially in photo-oxidation induced from ultraviolet radiation, temperature plays a very important role. Measuring the ultraviolet radiation during the exposure period is useful for comparison of the result of the exposure test, but it is not enough to compare the exposure results. Therefore, it is very important to find some index that can be used to evaluate the complex effect of received ultraviolet radiation and environmental temperature. The PERS is used to characterize the level of combined effect of ultraviolet radiation and temperature, and its characteristic proportionally increases depending on the UV radiation and temperature received.

3 Material

PERS is high-density polyethylene polymerized using molybdenum dioxide as a catalyst, containing the trans-form vinylene group. Other basic properties are as follows:

— absorbance ratio of trans-form vinylene group to methylene group: 1,0 to 1,3;

— melt flow rate (2,16 kg, 190 °C): 0,2 to 0,4 g/10 min;

— density: 950 to 965 kg/m^3;

— thickness: (0,2 ± 0,02) mm.

3.1 Preparation of PERS

After kneading for 5 min the material between two rolls whose surfaces are heated at 150 °C to 170 °C, cut into small pieces of 0,4 g to 0,5 g.

After pre-heating for 90 s in a compression moulding machine whose surface is heated at 160 °C to 180 °C, compress the material for 60 s, cool in a compression moulding machine whose surface temperature is 30 °C to 40 °C, for 60 s, and prepare the press sheet of the thickness mentioned above.

NOTE PERS can be obtained from the following organization:

Japan Weathering Test Center
1-3-7 Shibakoen Minatoku Tokyo Japan

4 Procedure

4.1 Method for measuring the carbonyl index of PERS

An infrared (IR) spectrophotometer should be used as the measuring apparatus.

Measure the infrared absorption spectra after irradiation, in the range of 2 200 cm^{-1} to 1 600 cm^{-1}. In this case, use for the scanning speed the same method as for a quantitative analysis.

The carbonyl index is determined in accordance with the following equation, based upon infrared absorbance spectra of exposed PERS. Absorbance at near 2 020 cm^{-1} peak is employed as an internal standard to correct for sample film thickness, while absorbance at near 1 715 cm^{-1} peak is used to indicate carbonyl group content. A typical diagram of IR absorbance is shown in Figure 1.

$$A_r = \frac{A_{1\,715}}{A_{2\,020}}$$

where

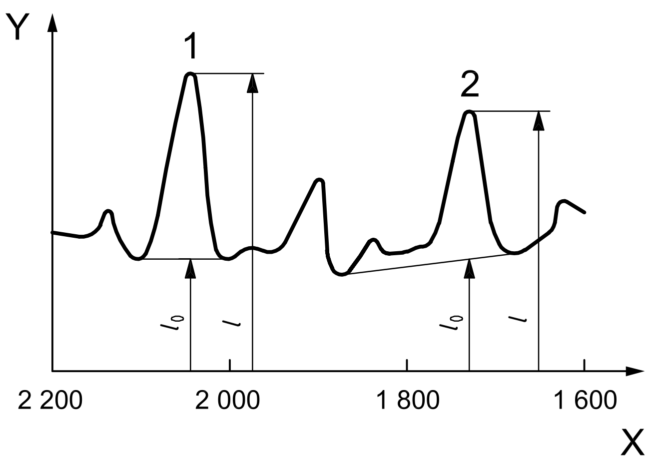
A_r is the absorbance ratio (carbonyl index);

$A_{1\,715}$ is the absorbance at near 1 715 cm^{-1} $(I - I_0)$;

$A_{2\,020}$ is the absorbance at near 2 020 cm^{-1} $(I - I_0)$;

I_0 is the absorbance measured by the base-line method at individual wave number;

I is the absorbance at the peak of individual wave number.



Key

Y absorbance

X wavenumber (cm^{-1})

1 peak near 2 020 cm^{-1}

2 peak near 1 715 cm^{-1}

Figure 1 — IR absorbance diagram showing base line and peak absorbance

4.2 Round Robin Test of laboratory light-source exposure devices with PERS

It is well known that the degradation of plastics materials or products used outdoors will occur by the combined effect of ultraviolet radiation and temperature, or other factors. It is also recognized that the reproducibility in the laboratory light-source exposure test will vary with the change in ultraviolet spectrum distributions and the chamber temperatures caused by deterioration of lamps and filters with operating time, even if ultraviolet radiation and black standard temperature (BST) or black panel temperature (BPT) are under constant conditions.

Since the PERS can quantitatively evaluate, as carbonyl index, the combined effect of UV radiation and temperature, the carbonyl index obtained can reflect changes in a given environment.

In order to verify the repeatability and reproducibility of the specimens and exposure test, the Round Robin Test (hereafter called RRT) using the PERS by laboratory light-source exposure devices in ISO/TC 61/SC 6/WG 2 was conducted.

4.2.1 Xenon-arc-lamp exposure

The test conditions were according to ISO 4892-2 [1]. The conditions are shown in Table 1. It was not requested to control the chamber temperature, but participants were requested to report this temperature.

Each participant was provided with 4 sets of PERS that were mounted in 150 mm × 70 mm plastics holders. One set of holders consists of 3 pieces of PERS.

Table 1 — Exposure conditions for xenon-arc lamp

Filter	daylight filter
Irradiance	0,5 W/(m^2·nm) at 340 nm or 60 W/m^2 (300 to 400 nm)
BST or BPT	(65 ± 3) °C for BST or (63 ± 3) °C for BPT
Chamber temperature	Arbitrary
Water spray	102 min of light only followed by 18 min of light plus water spray
Humidity	(50 ± 5) %
Period	24 h, 48 h, 72 h and 96 h

4.2.2 Open-flame carbon-arc-lamp exposure

The test conditions were according to ISO 4892-4 [2]. The conditions are shown in Table 2. The chamber temperature was not specified, but participants were requested to report this temperature.

Each participant was provided with 4 sets of PERS that were mounted in 150 mm × 70 mm plastics holders. One set of holders consists of 3 pieces of PERS.

Table 2 — Exposure conditions for open-flame carbon-arc lamp

Filter	Type 1 (Type 1 known as Corex 7058 filter)
BST or BPT	(65 ± 3) °C for BST or (63 ± 3) °C for BPT
Chamber temperature	Arbitrary
Water spray	102 min light only followed by 18 min of light plus water spray
Humidity	(50 ± 5) %
Period	24 h, 48 h, 72 h and 96 h

4.2.3 Fluorescence lamp exposure

The test conditions were according to ISO 4892-3 [3]. The conditions are shown in Table 3. Irradiance was not specified at any intensity.

Each participant was provided with 4 sets of PERS that were mounted in 150 mm × 70 mm plastics holders. One set of holders consists of 3 pieces of PERS.

Table 3 — Exposure conditions for fluorescent lamp

Lamp type	UVA340
Irradiance	Arbitrary
Mode	Mode 1: 4 h of dry UV exposure followed by 4 h of condensation
BPT	(63 ± 3) °C at UV exposure and (50 ± 3) °C at condensation
Period	8 h, 24 h, 32 h and 48 h

After each sample had been exposed for each exposure period, the carbonyl index was determined, based upon the method described in 4.1.

4.3 Outdoor exposure test of PERS

The result of the outdoor exposure test varies, even if it is conducted in the same place, because of differences due the seasonal climate changes. Although it is useful to measure the amount of ultraviolet radiation for comparison of exposure tests, it is not enough in the comparison only to consider the amount of ultraviolet radiation, because plastics are influenced not only by ultraviolet radiation but by temperature or by moisture. Since PERS is influenced by the combined effect of ultraviolet radiation and temperature, PERS were exposed in various places where the climate was different.

Six locations in different climates and different countries were selected: Sapporo, Choshi, Miyakojima (Japan), Serpong, Bandung (Indonesia) and Phoenix (USA). Locations and exposure angles are shown in Table 4.

Table 4 — Locations and exposure angles

Exposure site	Exposure angle	Latitude
Sapporo (Japan)	45° South	43° 03' N
Choshi (Japan)	30° South	35° 43' N
Miyakojima (Japan)	20° South	24° 44' N
Serpong (Indonesia)	5° [a] South and north	6° 15' S
Phoenix (USA)	34° South	33° 54' N
[a] From November to February, the samples face south; and from March to October, they face north.		

The conditions of the outdoor exposure test are based on ISO 877 [4]. Three pieces of PERS were exposed for 1 month. By replacing exposed PERS with new ones, the exposure test was repeated in the following months successively. The exposure test was repeated successively for more than 24 months.

The practical procedure of the outdoor exposure test is shown below.

1) The conditions of the outdoor exposure test are based on ISO 877.

2) Prepare a minimum of three PERS and expose them for 1 month. It is desirable to expose them at the beginning of a calendar month, in order to evaluate the condition of the month.

3) By replacing exposed PERSs with new ones, the exposure test is repeated in the following months successively.

4) The exposure test is repeated successively for at least 12 months.

5) The accumulated value of the carbonyl index for each month will be adopted for the index of combined effect of UV radiation and temperature at the site of exposure.

4.4 Consistency of laboratory light-source exposure devices

It is recognized that changes in the characteristics of lamps and filters with time, and changes in the chamber temperature of laboratory light-source exposure devices, influence the reproducibility and repeatability of test results, even if the test is operated under constant conditions of ultraviolet radiation and BST or BPT.

Since the carbonyl index of a polyethylene reference specimen is proportional to the environment where ultraviolet radiation and temperature are compounded, consistency of the exposure environment could be monitored with this reference material.

In order to verify the correct operation of a particular xenon-arc-lamp device, the control limit was determined by PERS.

The test conditions were according to ISO 4892-2. For different operating times of lamp and filters, exposure was repeated three times.

The practical procedure was carried out as follows.

1) The conditions of the laboratory light-source exposure test are based on ISO 4892.

2) PERS should be irradiated for about 100 h. The number of PERSs should not be less than three.

3) Carbonyl indices are measured after removal from exposure.

4) Repeat 1) to 3), at three different times.

5) Calculate the standard deviation “S” of each exposure.

6) Calculate the repeatability standard deviation “S_r”.

$$S_r = \sqrt{\frac{\sum S^2}{3}}$$

7) Calculate the control limit “CL” of this apparatus.

CL = total average value ± (2 × S_r)

5 Results and discussion

5.1 Result of RRT of laboratory light-source exposure devices with PERS

5.1.1 Xenon-arc-lamp exposure

Fourteen devices from thirteen organizations of five nations, Japan, the USA, Germany, Switzerland, and France, participated.

The test results are shown in Table 5.

Table 5 — Results of the round-robin test of xenon-arc-lamp exposure with PERS

Hours	Measurement		Lab. A	Lab. B	Lab. C	Lab. E	Lab. G	Lab. H	Lab. J-1	Lab. J-2	Lab. J-3	Lab. K	Lab. M	Lab. N	Lab. O	Lab. Q
24 (film 1, 2 and 3)	each lab.	mean	—	—	—	0,241	0,218	0,180	0,216	0,188	0,140	0,244	0,205	0,358	0,367	0,367
		std	—	—	——	0,011	0,005	0,020	0,011	0,008	0,013	0,008	0,008	0,005	0,010	0,012
		%	—	—	—	4,7	2,2	11,1	4,9	4,3	9,3	3,3	3,7	1,3	2,6	3,1
	ref. lab.	mean	0,202	0,310	0,214	0,256	0,214	0,189	0,222	0,209	0,149	0,242	0,226	0,377	0,463	0,355
		std	0,018	0,013	0,016	0,020	0,001	0,008	0,010	0,008	0,008	0,007	0,020	0,005	0,026	0,021
		%	8,7	4,2	7,5	7,9	0,3	4,3	4,3	3,9	5,4	2,9	8,9	1,3	5,5	6,0
48 (film 4, 5 and 6)	each lab.	mean	—	—	—	0,529	0,478	0,400	0,486	0,473	0,377	0,514	0,451	0,851	0,550	0,810
		std	—	—	—	0,014	0,008	0,010	0,026	0,070	0,012	0,024	0,008	0,038	0,022	0,026
		%	—	—	—	2,7	1,8	2,5	5,4	14,8	3,3	4,7	1,7	4,4	4,0	3,3
	ref. lab.	mean	0,510	0,659	0,492	0,552	0,488	0,393	0,543	0,520	0,395	0,533	0,488	0,879	0,665	0,824
		std	0,013	0,013	0,019	0,011	0,010	0,006	0,072	0,065	0,013	0,016	0,011	0,025	0,032	0,021
		%	2,6	2,0	3,9	2,0	1,9	1,4	13,2	12,5	3,2	3,0	2,2	2,8	4,7	2,6
72 (film 7, 8 and 9)	each lab.	mean	—	—	—	0,768	0,719	0,543	0,701	0,687	0,597	0,749	0,634	1,407	0,751	1,203
		std	—	—	—	0,029	0,041	0,006	0,009	0,024	0,058	0,012	0,022	0,056	0,031	0,055
		%	—	—	—	3,7	5,7	1,1	1,3	3,5	9,8	1,5	3,5	4,0	4,2	4,6
	ref. lab.	mean	0,688	1,039	0,723	0,779	0,705	0,530	0,728	0,731	0,641	0,767	0,697	1,456	0,885	1,234
		std	0,030	0,028	0,021	0,023	0,039	0,015	0,015	0,030	0,045	0,024	0,020	0,040	0,012	0,070
		%	4,3	2,7	2,9	3,0	5,5	2,9	2,1	4,1	7,1	3,1	2,9	2,8	1,4	5,7
96 (film 10, 11 and 12)	each lab.	mean	—	—	—	1,053	0,897	0,697	0,943	0,928	0,800	0,968	0,836	1,909	0,947	1,517
		std	—	—	—	0,031	0,070	0,023	0,060	0,022	0,006	0,017	0,034	—	0,037	0,107
		%	—	—	—	3,0	7,8	3,3	6,3	2,4	0,7	1,8	4,1	—	3,9	7,1
	ref. lab.	mean	0,882	1,351	0,953	1,058	0,922	0,689	1,010	0,990	0,845	0,961	0,906	1,969	1,090	1,516
		std	0,021	0,100	0,044	0,032	0,051	0,026	0,072	0,038	0,013	0,020	0,051	—	0,024	0,141
		%	2,4	7,4	4,6	3,0	5,6	3,7	7,1	3,9	1,6	2,0	5,6	—	2,2	9,3

each lab.: carbonyl index measured by each laboratory.

ref. lab.: carbonyl index measured by reference laboratory.

std.: standard deviation.

%: percentage of standard deviation to mean value.

After finishing the exposure test, specimens were measured to calculate carbonyl indices at each laboratory, and returned to the reference organization (Japan Weathering Test Center). In this reference organization, the characteristic values were measured by a single operator with a single item of equipment.

As shown in Table 5, the data of the reference laboratory showed the same tendency as the data of each laboratory. Mean values and standard deviations of each laboratory and of the reference laboratory were almost the same. This means that even if the position of the films, measurement apparatus, and operators are different, almost the same results can be obtained.

Each laboratory's data were plotted together with those of the reference laboratory in Figure 2. All data showed linear relations between carbonyl indices (CI) and testing hours. The correlation coefficients were 0,95 or more.

The slope showing the relation between testing hours and CI differed between items of equipment, and this is collectively indicated in Figure 3. Eight of 14 different items of equipment demonstrated slopes in a range from 0,009 to 0,011, another two were less than 0,009 and the remaining four were more than 0,011.

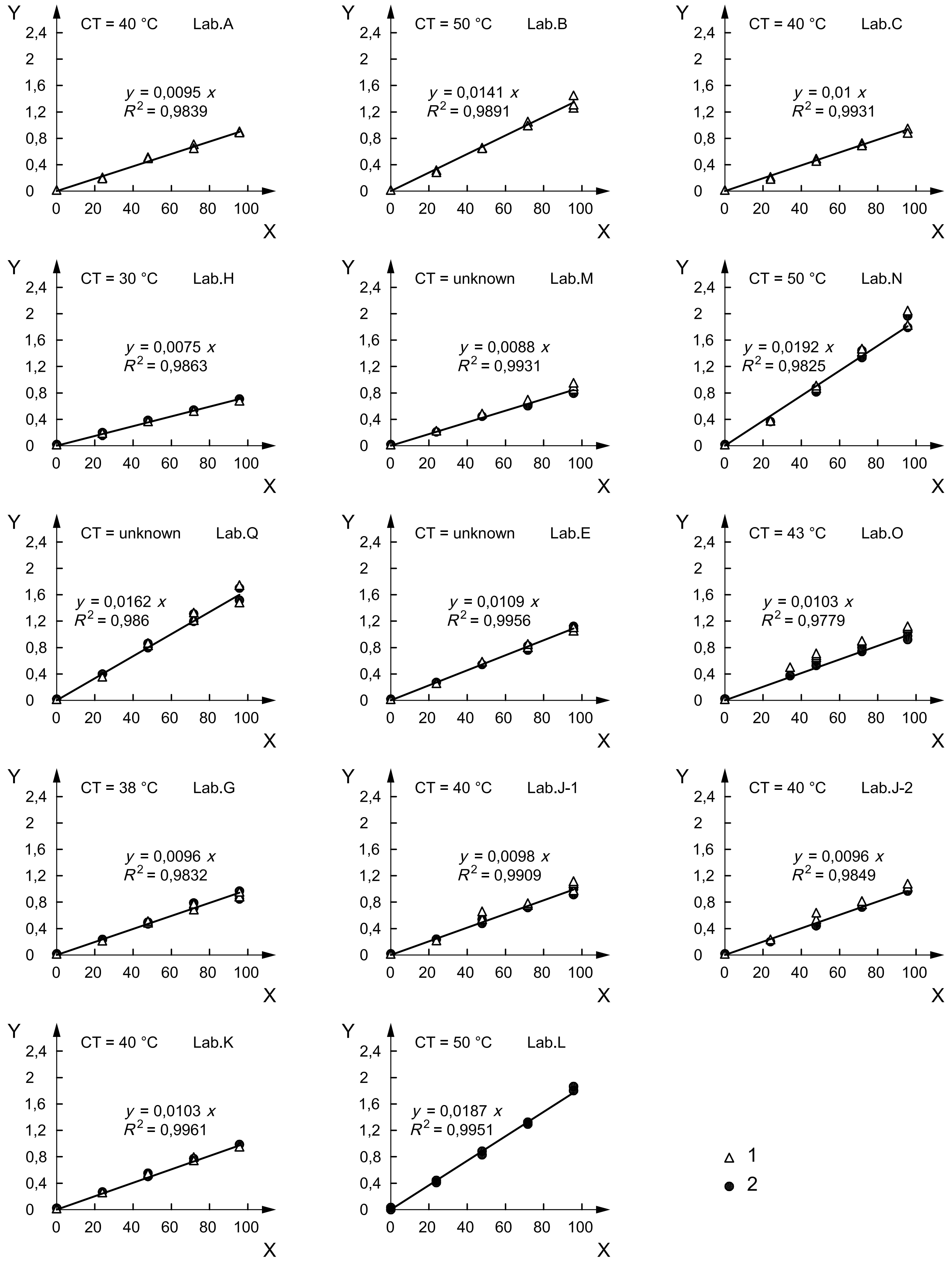
On the other hand, the relation between slopes and chamber temperatures was examined. As a result, the temperatures of eight items of equipment ranged from 38 °C to 43 °C, and the temperature of the equipment having the maximum slope was as high as 50 °C, as opposed to the equipment with the minimum slope indicating 30 °C.

From this fact, the chamber temperature has a great influence on the slope. If the chamber temperature is not kept constant, even though items of equipment are controlled with a UV radiometer and BST or BPT, the result obtained would vary (see [5] in the Bibliography). Thus, a quantitative indication of exposure environments obtained through PERS is vital for the reproducibility of the experiment.

The Arrhenius plot of the chamber temperature and slope is shown in Figure 4. The activation energy calculated was about 35 kJ/mol, which was consistent with previous studies (see [6] and [7] in the Bibliography). All these data include BPT and BST and the higher point also had a higher BPT. PERS is susceptible to chamber temperature, therefore, if PERS was used, it is able to indicate the different chamber conditions.

The results of analysis of variance in data from all laboratories participating in the RRT are shown in Table 6. The variances between laboratories were about 98 %, so this means that the difference of the test results was attributable to exposure itself.

Although this RRT unified test conditions of UV intensity and BST or BPT, the chamber temperatures were different among laboratories, and therefore, different slopes were mainly due to differences in the chamber temperatures of equipment.



Key

Y carbonyl indices

X testing hours

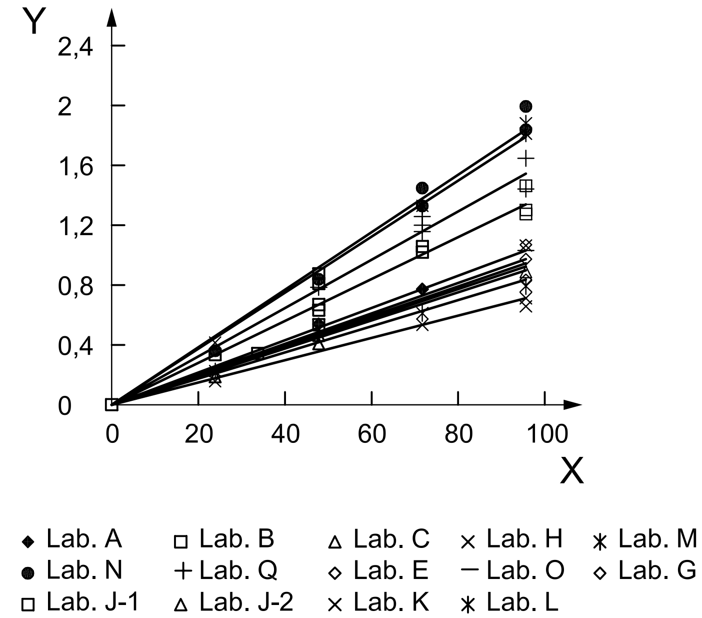
1 reference laboratory

2 each laboratory

CT chamber temperature

R^2 determination coefficient

Figure 2 — The round-robin test of PERS in xenon-arc exposure apparatus

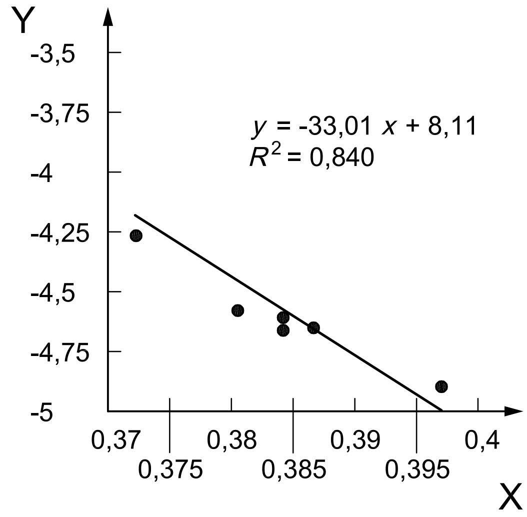


Key

Y carbonyl indices

X testing hours

Figure 3 — Summary of xenon-arc exposure



Key

R^2 determination coefficient

X I/*RT* (*RT* gas constant × chamber temperature)

Y LN*K* logarithm of slope *K* of regression line

Figure 4 — Relationship between chamber temperature and degradation speed

Table 6 — Result of analysis of variance of xenon-arc-lamp exposure

Exposure hours	Participating laboratory measurement			Reference laboratory measurement		
	Sum of squares		% of total variance	Sum of squares		% of total variance
24	between lab.	0,214	98,2	between lab.	0,291	98,0
	within lab.	0,004	1,8	within lab.	0,006	2,0
	total	0,218	100,0	total	0,297	100,0
48	between lab.	0,759	97,4	between lab.	0,800	96,9
	within lab.	0,020	2,6	within lab.	0,026	3,1
	total	0,779	100,0	total	0,826	100,0
72	between lab.	2,331	98,6	between lab.	2,457	98,8
	within lab.	0,033	1,4	within lab.	0,031	1,2
	total	2,364	100,0	total	2,487	100,0
96	between lab.	3,392	97,5	between lab.	3,394	96,8
	within lab.	0,086	2,5	within lab.	0,114	3,2
	total	3,478	100,0	total	3,508	100,0

5.1.2 Open-flame carbon-arc-lamp exposure

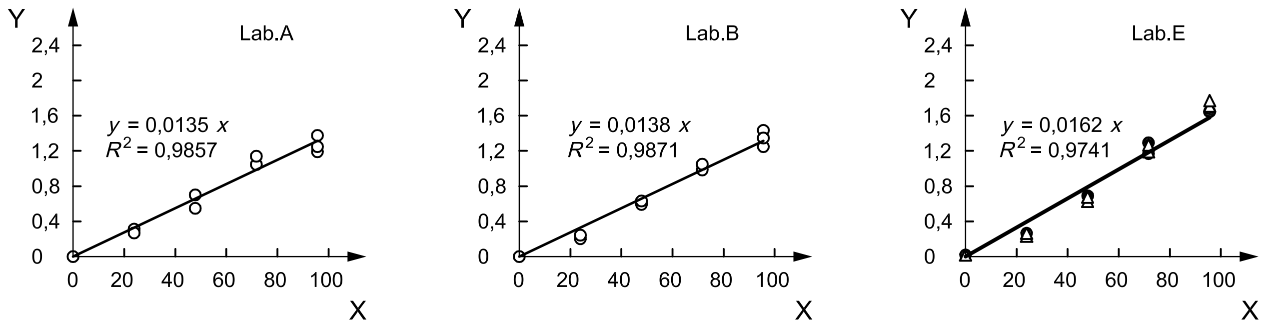
Three devices from three organizations of two nations, Japan and the USA, participated.

The result of open-flame carbon is shown in a Figure 5. Three organizations participated. The data from every organization indicated linearity and a correlation coefficient of 0,95 or more. In these three devices, the reported BPT was the same. Therefore, it is thought that it reflects the difference of chamber temperature. The slope is larger than for the xenon-arc lamp. It is based on difference of light intensity, difference of spectral distribution, difference of chamber temperature, etc.

5.1.3 Fluorescent lamp exposure

Five devices from four organizations of three nations, Japan, the USA and Switzerland, participated.

Figure 6 represents the results of an ultraviolet fluorescent light test in which five organizations participated. For the test conditions, a repeat of a cycle composed of 4 h irradiation and 4 h darkness was used, which means that the period when UV is irradiated is half of that for other light sources. The results show that the linearity is not as good as for other light sources, and the data after 24 h exponentially increased despite the half of UV irradiation. The reason that this phenomenon occurred is because temperatures in the chamber were kept at about 50 °C during darkness, whereby a reaction in PERS proceeded. Thus, in this case, it cannot be said that it is proportional to the exposure environment.



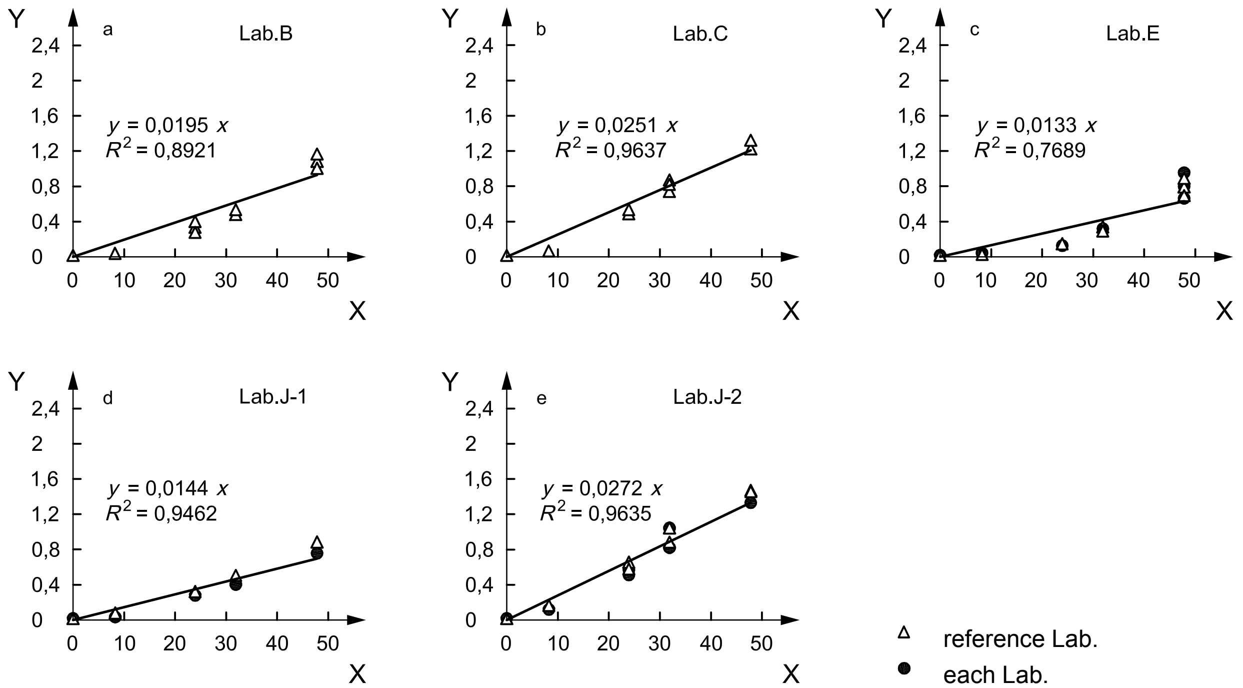
Key

Y carbonyl indices

X testing hours

R^2 determination coefficient

Figure 5 — The Round Robin Test of PERS in open-flame carbon-arc exposure apparatus



Key

Y carbonyl indices

X testing hours

R^2 determination coefficient

[a] UV intensity is unknown.

[b] 0,76 W (m^2.nm) at 340 nm.

[c] UV intensity is unknown.

[d] 0,77 W (m^2.nm) at 340 nm.

[e] 0,63 W (m^2.nm) at 313 nm.

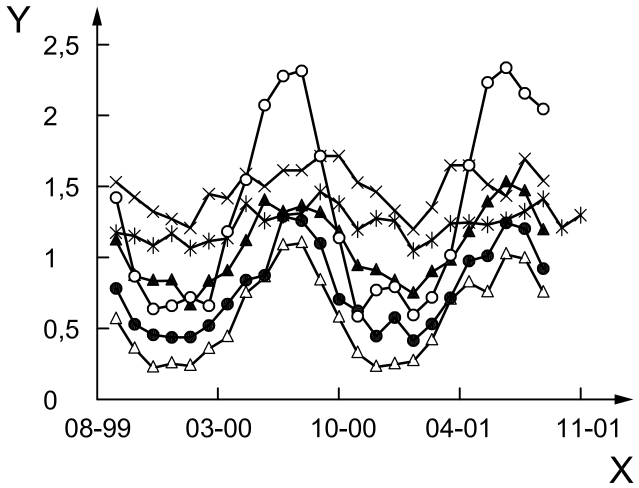
Figure 6 — Round-Robin Test of PERS in fluorescent-UV-lamp exposure apparatus

5.2 Characterizing the conditions of outdoor exposure test site

The results of the outdoor exposure test on PERS carried out in six places, Sapporo, Choshi, Miyakojima (Japan), Serpong, Bandung (Indonesia), and Phoenix (USA), are shown below. Figure 7 shows carbonyl indices calculated from PERS exposed every month, and Figure 8 shows the cumulative sum of them.

These values reflect the difference of the climate of the sites where the exposures took place. Serpong is in the tropics where the climate change is rather small throughout the year. In contrast, the differences between summer and winter are quite pronounced in the other places. The cumulative value of CI recorded from Serpong is relatively linear due to the small climate change, while those recorded in the other areas have a wavy character reflecting seasonal variation.

From Figures 7 and 8, it is evident that the carbonyl index reflects definitely the climatic features of the sites where the exposures were conducted.



Key

Y carbonyl index

X months

—△— Sapporo (Japan)

—●— Choshi (Japan)

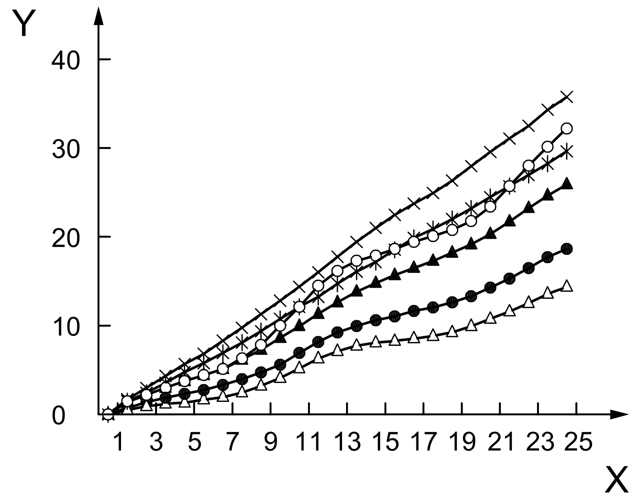
—▲— Miyakojima (Japan)

—×— Serpong (Indonesia)

—○— Phoenix (USA)

—✳— Bandung (Indonesia)

Figure 7 — Behaviour of monthly carbonyl index at each place



Key

Y accumulated carbonyl index

X accumulated months

—△— Sapporo (Japan)

—●— Choshi (Japan)

—▲— Miyakojima (Japan)

—×— Serpong (Indonesia)

—○— Phoenix (USA)

—✳— Bandung (Indonesia)

Figure 8 — Cumulative sum of monthly carbonyl index at each place

5.3 Examples of correlation between outdoor exposure test and laboratory light-source exposure test using PERS

The degree to which plastics materials are damaged in outdoor environments is influenced by the UV radiation, temperature, humidity, etc., that they received. Of these factors, in photo-oxidation caused by UV, temperature in particular plays an important role.

In evaluating the weatherability of plastics, it is often found that it is not possible to correlate the outdoor exposure test with the laboratory light-source exposure test for only the amount of UV received. This indicates the need for some index indicating combined effects based on received UV and temperature.

The test method described here is one that uses carbonyl indices, as an index showing combined effects, determined from carbonyl group linearly increasing in PERS, depending on received UV and temperature. As shown in Figure 2, the carbonyl index of PERS is proportional to the exposure period, and as shown in Figure 7, it reflects the seasonal change of climate in an outdoor environment. Thus, from these results, the carbonyl index can be used as the index exhibiting the environmental condition of the laboratory light-source exposure test and the outdoor exposure test mainly dominated by UV and temperature.

Table 7 shows examples of the correlation between outdoor exposure and xenon-arc exposure. The exposure was carried out according to the methods described in 4.2 and 4.3. When the carbonyl index obtained from outdoor exposure and xenon-arc exposure is the same, it is considered that these exposures are equivalent.

This value includes the effect of temperature. Since the temperature in the chamber of a laboratory light-source exposure device is higher than that outdoors, the time to become equivalent to a cool climate area like Sapporo (Japan) becomes short, and the time to become equivalent to a high temperature area like Phoenix (USA) becomes long.

The degradation mechanism of PERS is an auto-oxidation reaction caused by ultraviolet irradiation. In many cases, plastics materials also degrade, mainly by photo-induced auto-oxidation in outdoor environments. Therefore, when the temperature promotes the diffusion rate of the substances such as oxygen in the rate-determination stage, it is thought that the carbonyl index correlates with the oxidation rate of other plastics materials. Changes in experimental properties that originated in the decrease in molecular weight result from a photo-induced auto-oxidation reaction, like gloss loss or elongation of tensile test, often correlate with the carbonyl index (see [8] and [9] in the Bibliography).

Table 7 — Comparison between outdoor exposure and xenon-arc exposure for PERS

Exposure site	Cumulative carbonyl index value of 1 year [a]	UV radiant exposure at exposed angle [a] (MJ/m^2) (315 to 400 nm)	Average temperature of 1 year [a]	Equivalent hours of xenon exposure [b] to 1 year outdoor exposure based on the comparison of carbonyl index
Sapporo, Japan	7,2	300 [c]	9,3	700 to 800 [d]
Choshi, Japan	9,3	336	14,9	900 to 1 100
Miyakojima, Japan	12,7	377	23,4	1 300 to 1 500
Phoenix, USA	16,1	434 [c]	22,7	1 600 to 1 900

[a] Observed value from October 1999 to September 2000.

[b] Test condition: Irradiance level 60 W/m², BPT (63 ± 3) °C, 18 min spray/120 min.

[c] The Sapporo value is converted from the solar radiation, and the Phoenix value is 1,3 times the value obtained by actual measurement to allow for a different UV range.

[d] The minimum and maximum values are based on the value of the equipment given in 4.4. This value changes with each item of equipment as in 5.1.

This correlation, however, is not applicable simply to all kinds of plastics materials. Certain relations can only be expected if the influence of the degradation factor on carbonyl generation of PERS, and the influence of the degradation factor on the change in properties of interested materials, are either equivalent or their effect on the material is known.

5.4 Control limit of particular laboratory light-source exposure apparatus

An example of the control limit of the equipment is shown in Table 8 and Figure 9, from the results of 3 times the 120 h xenon exposure. How to calculate these values is described in 4.4.These were conducted in the same conditions and using the same machine at different times.

The difference between the results for three times the exposure is based on characteristic change of the lamp and filters with the lapse of time and different chamber conditions, like temperature or wind velocity, etc.

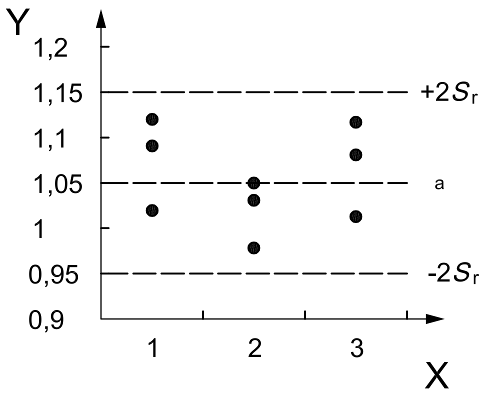
If the reference specimen is sometimes exposed and the carbonyl index exists in this range, it can be said that the exposure device is correctly operated or, if it exists outside the range, the reasons for the discrepancy must be explained and resolved.

However, use of the PERS is supplementary to the controls provided by the ultraviolet radiometer, BST, or chamber air temperature. Since PERS is susceptible to a change of chamber temperature, it should not be used to calibrate the ultraviolet radiometer.

Table 8 — Example of calibration of xenon-arc apparatus

Times	1	2	3	Average	Standard deviation
1	1,091	1,119	1,019	1,076	0,052
2	0,978	1,030	1,052	1,020	0,038
3	1,013	1,080	1,116	1,070	0,052
			Total average	1,055	
				Repeatability standard deviation S_r	0,048

Repeatability limit = total average ± 2 × S_r = 1,055 ± 0,096 (for 120 h)



Key

X times

Y carbonyl indices

[a] Total average.

Figure 9 — Example of control chart of laboratory light-source exposure device

6 Conclusion

6.1 Results of RRT

1) A linear correlation between exposure period and increase of carbonyl index is shown for all xenon and open-flame carbon devices in all participating laboratories in the RRT.

2) Differences of increasing rate of carbonyl index among xenon and open-flame carbon devices participating in the RRT are mainly influenced by chamber temperatures under UV intensity and BST or BPT in constant conditions.

3) Measurement results of the same samples between the reference laboratory and participating laboratories were almost the same. It means that, even if the position of the films, measurement apparatus, and operators are different, almost the same results can be obtained.

4) The variances between laboratories were about 98 %, so this means that the difference of the test results was attributable to exposure itself.

5) When the irradiation and darkness were repeated, the oxidation reaction also occurred at a dark period in the fluorescent lamp exposure.

6.2 Outdoor exposure of PERS

The carbonyl index of PERS can reflect the seasonal climate change in many places. Therefore, with the carbonyl index measured for each PERS, the accumulated value of the carbonyl index for each month should be adopted as the index of combined effect of UV radiation and temperature at the site of exposure.

6.3 Correlation between outdoor and xenon-arc-lamp exposure for PERS

The correlation between outdoor exposure and xenon-arc exposure by PERS has been obtained. The time to become equivalent to a cool climate area like Sapporo (Japan) becomes short, and the time to become equivalent to a high temperature area like Phoenix (USA) becomes long.

6.4 Consistency of laboratory light-source exposure devices

Supplementary tests using PERS can be used to control the laboratory light-source exposure devices provided by the ultraviolet radiometer, BST, or chamber air temperature.

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