

# Combined impact of UV radiation and nitric acid on high-density polyethylene containers as a laboratory test

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## Abstract

In a laboratory test, transparent high-density polyethylene (HDPE) jerrycans have been exposed to both UV radiation and 55 wt-% nitric acid solution at  $(41 \pm 2)^\circ\text{C}$ , for up to 20 days. For comparison, UV radiant exposure (21 days) and nitric acid exposure (up to 6 weeks) were performed separately, at nearly equal temperatures. The damages are compared with FTIR spectroscopy in ATR and HT-gel permeation chromatography (GPC) on a molecular level and with hydraulic internal pressure testing as a component test. For the used jerrycans, relevant oxidation can only be found after the combined exposure. This is caused by the decomposition of nitric acid into nitrous gases by UV radiation, which is also observed at lower concentrations (28 wt-%). After 6 days of laboratory exposure, this is rated as critical, which corresponds to about 1/10 year in Central Europe, according to the UV radiant exposure. The gradual increase in oxidative damage shows the reproducibility of the test.

## KEYWORDS

high-density polyethylene, molecular mass distribution, nitric acid, UV radiation

## 1 | INTRODUCTION

A variety of containers made from high-density polyethylene (HDPE) can be used for transport and storage of dangerous goods. For this purpose, the European Agreement concerning the International Carriage of Dangerous Goods by Road ADR<sup>1</sup> has been established, which includes requirements for the construction and compatibility testing of plastic packaging. Here, 55 wt-% nitric acid solution plays a particular role, as according to ADR clause 6.1.6 e, it can be used as a standard liquid for verifying the chemical compatibility testing of polyethylene packaging, for all substances having an oxidizing effect on PE.

However, it was shown<sup>2</sup> for several transparent HDPE jerrycans that a simple storage of the 55% nitric acid ( $\text{HNO}_3$ -55%) does not necessarily lead to oxidation. Instead, it was found that the UV radiation transmitted through the HDPE can decompose the filling good<sup>3</sup>

and then the resulting decomposition products damage the HDPE (secondary damage).

In outdoor exposure tests,<sup>2</sup> it was found that a damaging action of the  $\text{HNO}_3$ -55% only occurs in combination with its decomposition, caused by UV radiation. The dissolved  $\text{HNO}_3$  decomposes into gaseous nitrogen oxides ( $4 \text{HNO}_3 \leftrightarrow 2 \text{H}_2\text{O} + 4 \text{NO}_2 + \text{O}_2$ ). Nitrogen dioxide  $\text{NO}_2$  is a free radical that can act as an autoxidation initiator<sup>4</sup> in HDPE, leading to strong oxidation of the polyethylene (PE) molecules through the formation of carbonyl groups.<sup>5</sup>

Outdoor exposures, as in Geburtig et al.,<sup>2</sup> have a bad reproducibility as well as several safety challenges. Especially because of the strong temporal variability of the weather, they are not suited for extensive systematic further investigations. Therefore, a device was developed to investigate the combined impact of UV radiation and dangerous liquids under safe conditions within a lab, for medium scale containers.<sup>6,7</sup> Within this device, the exposure parameters can be

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adopted to maximum conditions from practice, skipping irrelevant timesteps, such as night times.

In the following, this device is used to identify the critical exposure durations for a specific type of jerrycan, filled with HNO<sub>3</sub>-55%. The problems after combined exposure demonstrated in outdoor exposure<sup>2</sup> can now be replicated in the laboratory, with the advantages of repeatability and the ability to control and adjust parameters.

## 2 | MATERIALS, EXPOSURES AND METHODS

### 2.1 | Materials

An unpigmented HDPE was used (Hostalen ACP 5531B), in the form of 5-L jerrycans, produced at Kautex Textron GmbH Co. KG, with expired approval (UN 3H1/Y1,9/170/13/D/BAM 11860). Meanwhile the jerrycans had been stored at room temperature in darkness. The wall thickness was about 1 mm. The UV transmittance at 300 nm (near global solar radiation cut-on) was still about 20%, so that a considerable amount of UV radiation acts on the filling good.

Nitric acid was generally used in 55 wt-% concentration, except for one experiment with 28 wt-%.

### 2.2 | Exposures

An overview of the exposures carried out is given in Table 1.

The device for the combined exposure has been described before.<sup>6,7</sup> The acid-filled jerrycan was placed on a slowly rotating table. The filling height was 5 cm (about 500 ml), because previous tests had shown that the damage occurred mainly in the gas phase. The jerrycan was irradiated from two sides with out of a field of lamps, respectively. UVA-340 nm lamps, according to ISO 4892-3, UVA340, Type 1A, were used, which mimic the cut-on of global radiation very well. Due to the potentially dangerous gases, the device was placed under a fume cupboard. The unit is not actively temperature controlled. Instead, the temperature was recorded via a data logger.

To ensure the best possible comparability, for UV exposure only, the jerrycans were exposed in the same device filled with water instead of nitric acid. With a UV irradiance of the UVA-340 nm lamps of 25 W/m<sup>2</sup> from two sides, 21 days correspond approximately to the UV radiant exposure of 1/3 year in Central Europe.

The 40°C storage of HNO<sub>3</sub> was carried out in a heated chamber, in accordance with the usual dangerous goods tests. To check possible effects of re-drying, the samples were reconditioned differently after their 6-week exposure to HNO<sub>3</sub>: S06: 14 days vacuum, S12: 8 days, and S11: 2 h (all at 23°C). All other jerrycans had been reconditioned at 23°C for several days.

The jerrycan S10 was irradiated for 5 h before being removed to the dark 40°C heat chamber to check the decomposition conditions.

The jerrycan S13 was exposed to the combined exposure at half the HNO<sub>3</sub> concentration.

**TABLE 1** Exposures carried out

| Sample | Exposure                    | Duration         | T / °C |    |
|--------|-----------------------------|------------------|--------|----|
| S14    | original                    | 0d               |        |    |
| S15    |                             | 0d               |        |    |
| S20    | UV                          | 20.9d            | 39.3   |    |
| S07    | HNO <sub>3</sub> + UV       | 1.0d             | 42.5   |    |
| S04    |                             | 2.9d             | 42.0   |    |
| S16    |                             | 5.9d             | 41.8   |    |
| S09    |                             | 8.9d             | 39.8   |    |
| S03    |                             | 13.0d            | 41.4   |    |
| S05    |                             | 13.0d            | 42.2   |    |
| S08    |                             | 18.9d            | 41.1   |    |
| S02    |                             | 19.9d            | 42.1   |    |
| S06    |                             | HNO <sub>3</sub> | 42.0d  | 40 |
| S12    |                             |                  | 41.9d  | 40 |
| S11    | 41.9d                       |                  | 40     |    |
| S17    | 20.9d                       |                  | 40     |    |
| S18    | 8.0d                        |                  | 40     |    |
| S19    | 1.0d                        | 40               |        |    |
| S10    | HNO <sub>3</sub> + UV       | 5 h              | 39.0   |    |
|        | HNO <sub>3</sub>            | 5d               | 40     |    |
| S13    | HNO <sub>3</sub> (28%) + UV | 12.9d            | 40.7   |    |

For all exposures, the temperature was in a comparable range of (41 ± 2)°C.

### 2.3 | Methods

#### 2.3.1 | Hydraulic internal pressure test

A hydraulic internal pressure test was carried out for all jerrycans after the respective exposure.<sup>8</sup> This test is very close to a typical mechanical container stress in later use. The jerrycan lid was equipped with two valves, one for the water inlet and a second for the pressure sensor. By controlled water supply ([23 ± 1]°C), the overpressure was regulated to a constant value of (1.70 ± 0.02) bar, according to the approval data. If this test pressure could be maintained for 1 h, the pressure was further increased gradually until failure, in contrast to the test requirements in the United Nations.<sup>1</sup> Time dependent mass and pressure were recorded.

#### 2.3.2 | Colour measurement

The colour was measured with a Minolta CM 2600d spectrophotometer from Konica Minolta Europe GmbH, Langenhagen, Germany (6-mm aperture, 8/d geometry, SCI, D65, 10° observer) and characterized as CIE Lab values. White paper was used as backing.

### 2.3.3 | FTIR/ATR spectroscopy

A Nexus 470 Fourier transform infrared spectrometer from Nicolet Instruments (Germany) with a Smart-Orbit diamond ATR accessory and a DTGS-KBr detector with diamond crystal was used. Spectra in the range 4000 to 600  $\text{cm}^{-1}$  were obtained by averaging 32 scans with a resolution of 4  $\text{cm}^{-1}$ . The spectra shown were taken of the potentially more damaged surface of the jerrycans (inner surface i in case of  $\text{HNO}_3$  use, outer surface o for UV and water). For each jerrycan, samples were taken from the bottom of one side wall (L, liquid phase) as well as from the middle of the side wall (G, gaseous phase).

### 2.3.4 | Gel permeation chromatography (GPC)

A high-temperature GPC equipped with infrared (IR4) and viscosity detection (PolymerChar GPC-IR), an isocratic HPLC pump (PSS-Security) and an autosampler from PolymerChar GPC-IR running at 160°C were used. The eluent used was 1,2,4-trichlorobenzene stabilized with 0.1% BHT. The flow rate was 1.0 ml/min. This analytical method describes the determination of the molar mass distribution of poly(ethylene) after gel chromatographic separation on GPC columns (3 × PSS POLYOLEFIN linear XL, 30 cm × 0,8 cm, 10- $\mu\text{m}$  pore size). This method is a relative method. The molar masses are calculated based on a universal calibration with 12 poly(styrene) standards from Polymer Standards Service GmbH, in the range of  $M_p$  (1.2 × 10<sup>7</sup>–266 g/mol) by conversion according to Mark-Houwink parameters.

The 200  $\mu\text{l}$  of an approximately 2 mg/ml (8-ml total) solution in the eluent (internal filtration unit) were injected. The error range was estimated at approximately  $\pm 10\%$ .

## 3 | RESULTS AND DISCUSSION

### 3.1 | Exposure

An orange coloration during exposure indicates the formation of nitrous gases and occurred in principle with all jerrycans exposed in

combination, even when  $\text{HNO}_3$ -28% was used (S13). Apparently, this decomposition of the nitric acid, which was also observed in the transparent jerrycans during outdoor exposure,<sup>2</sup> is caused by the UV range of solar radiation. In jerrycan S10 (with  $\text{HNO}_3$ -55%), which was repositioned from the UV chamber to the dark heating chamber after 5 h, this orange colour disappeared, indicating that in the absence of UV irradiation the reverse reaction of the  $\text{HNO}_3$  decomposition takes place.

### 3.2 | Hydraulic internal pressure test

Figure 1 shows typical mass and overpressure curves, after various exposures. Since an early failure was to be expected with combined stress, the target pressure was approached in steps of 0.5 bar.

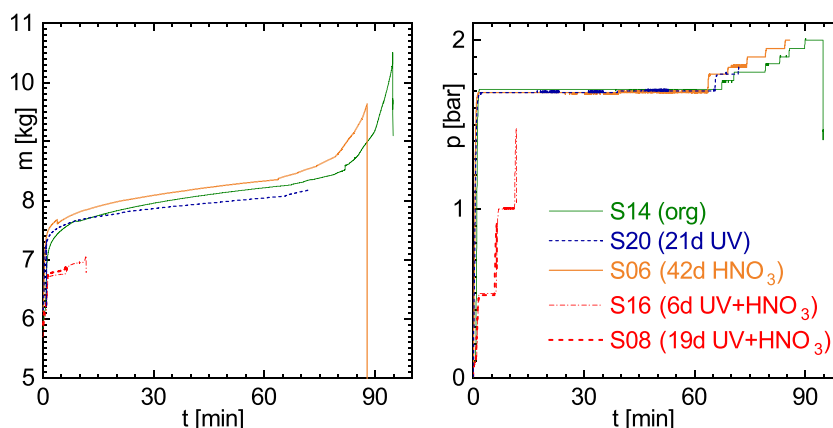
In fact, the target pressure was not reached after only 6 days of combined exposure, see Figure 2. From this exposure duration onwards, the failure occurred as brittle cracking. In contrast, all jerrycans that withstood the target pressure showed ductile failure. This was characterized by plastic flow, which could be observed as the formation of stripe-like thinnings which then hardened. The jerrycan grows into an almost spherical balloon and holds up to 170% of its original volume. Such visco-plastic deformation behaviour in combination with the ductile failure was also observed by Menrad,<sup>8</sup> after 21 days prestoring at 40°C with specific standard liquids, among others  $\text{HNO}_3$ -55%.

The observed change from ductile to brittle failure is a clear indication of oxidation after combined exposure.

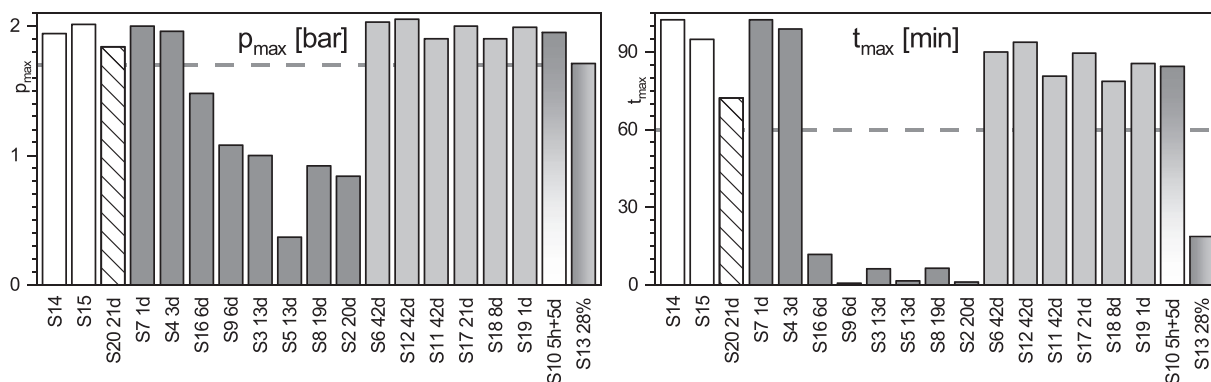
The duration of the re-drying of the jerrycans had no influence on the results of the internal pressure test.

### 3.3 | Colour change

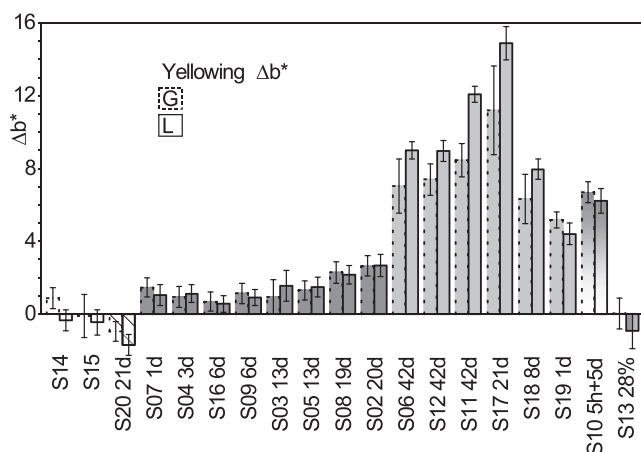
Clear yellowing of the HDPE only occurred when the nitric acid was allowed to act on the jerrycan in an undissociated state (without UV radiation), as shown in Figure 3. This seems to be in contrast to the observations after outdoor exposure,<sup>2</sup> but also at that time the nitric



**FIGURE 1** Time logged mass  $m$  and overpressure  $p$  during hydraulic internal pressure test, after various exposures



**FIGURE 2** Maximum of gauge pressure  $p_{\max}$  (left) and time till leakage  $t_{\max}$  of hydraulic internal pressure test, for all exposures. The target pressure should be able to be maintained for 60 min (dashed lines)



**FIGURE 3** Measured yellowing  $\Delta b^*$ , after the respective exposure

acid had exposure durations without UV radiation, at least before emptying. A comparison of S06, S12 and S11 indicates that drying reduced the yellowing. The measured changes in the lightness value L were not meaningful. The fact that a clear white coloration was not observed, as was the case there,<sup>2</sup> may have been due to the lack of the black background during the colour measurement. The differences in yellowing could be due to chromophoric compounds, which are not photostable themselves.

### 3.4 | FTIR/ATR spectroscopy

FTIR/ATR spectra after different exposures are shown in Figure 4. After  $\text{HNO}_3$  treatment, the measurements were taken from the inner sides (i) of the jerrycans. All measurements in Figure 4 refer to the gas phase (G), above the filling level.

For the unaged original (S14), the typical PE bands occurred at 1473, 1463, 1366, and 1351  $\text{cm}^{-1}$ , respectively,<sup>9</sup> as well as the

amorphous bands at 1080 and 1303  $\text{cm}^{-1}$ .<sup>10</sup> The peak at 965  $\text{cm}^{-1}$  was assigned to the vinylene group.<sup>11</sup>

For the S20 jerrycan (21 days of UV exposure), the ATR spectrum was taken from the outer side (o). Here, compared to the original, a small peak appeared at 909  $\text{cm}^{-1}$ , which was assigned to the vinyl group.<sup>11</sup> Only a small peak hinted in the carbonyl band region around 1713  $\text{cm}^{-1}$ ,<sup>11</sup> which shows that hardly any oxidation has taken place.

After 6 weeks of  $\text{HNO}_3$  exposure (S06), small bands at 1629, 1275, 860, and rudimentary at 1648  $\text{cm}^{-1}$  appeared compared to the original, which were assigned to various nitrates- $\text{ONO}_2$ .<sup>12</sup>

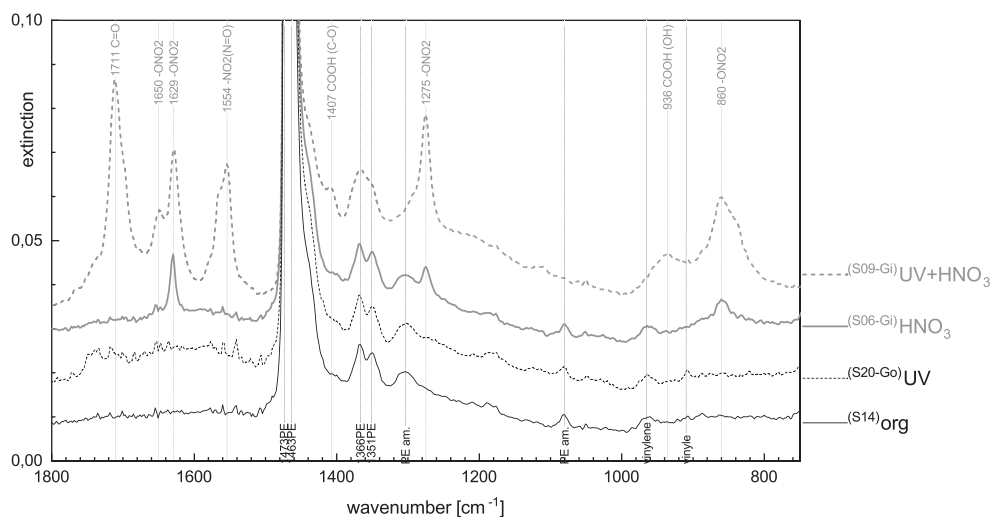
After combined exposure (S09, 9 days), these bands were more pronounced. Furthermore, bands appeared at 936  $\text{cm}^{-1}$ , 1407  $\text{cm}^{-1}$ , and a very strong band at 1711  $\text{cm}^{-1}$ , revealing carbonyl formation from either ketones or carbonic acids.<sup>5,13</sup> A further strong peak at 1554  $\text{cm}^{-1}$  could be attributed to the N=O-bond in a nitro group.<sup>5</sup> At the same time, the amorphous PE bands at 1303 and 1080  $\text{cm}^{-1}$  were reduced, indicating an increased degradation of the amorphous structures.

These FTIR measurements agree very well with those after outdoor weathering,<sup>2</sup> showing equal material behaviour at the respective exposures.

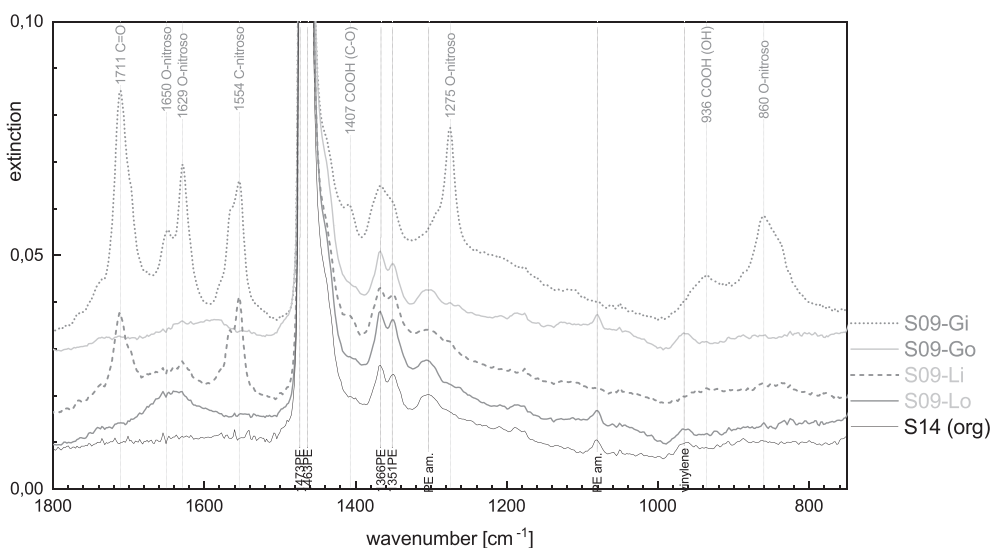
Typical FTIR spectra from various positions of one jerrycan are shown in Figure 5, after 9 days combined exposure (S09) in comparison to an unexposed specimen (S14). On the outer sides (o), the IR bands did not change compared to the original. On the inner sides (i) clear bands at 1554 and 1711  $\text{cm}^{-1}$  appeared in the liquid phase (L) as well as in the gas phase (G). Also, the reduction of the amorphous PE bands at 1303 and 1080  $\text{cm}^{-1}$  were noticeable both in the liquid and gaseous phase. Further bands at 860, 1275 and 1629  $\text{cm}^{-1}$  were only formed in the gas phase. This confirms the theoretical considerations of Oluwoye et al,<sup>14</sup> who claim that the addition of nitric oxide and nitrogen dioxide should result in the formation of C-nitroso and nitro species, respectively, and that the presence of molecular oxygen would sustain the formation of O-nitroso compounds, organic nitrites and nitrates.

Re-drying the jerrycans did not change the IR spectra.

**FIGURE 4** FTIR/ATR spectra after various exposures



**FIGURE 5** FTIR/ATR spectra of different positions of jerrycan S09, compared to the original S14



For more quantitative consideration, a carbonyl index CI was defined as the peak height at  $1711\text{ cm}^{-1}$  above a local baseline. Figure 6 shows the CI values for different exposures and durations. The combined exposures (filled squares) carried out in random order show a uniform increase in oxidation with duration, which confirms the reproducibility of the laboratory exposure. After 13 days with  $\text{HNO}_3$ -28%, the carbonyl formation corresponds approximately to that of 3 days with  $\text{HNO}_3$ -55%. The pure UV exposure hardly showed any oxidation.

### 3.5 | Molar mass distribution

The molar mass distribution was examined for selected samples, see Figure 7. Table 2 lists the corresponding number average  $M_n$ , the weight average  $M_w$  and the curve maximum  $M_p$ .

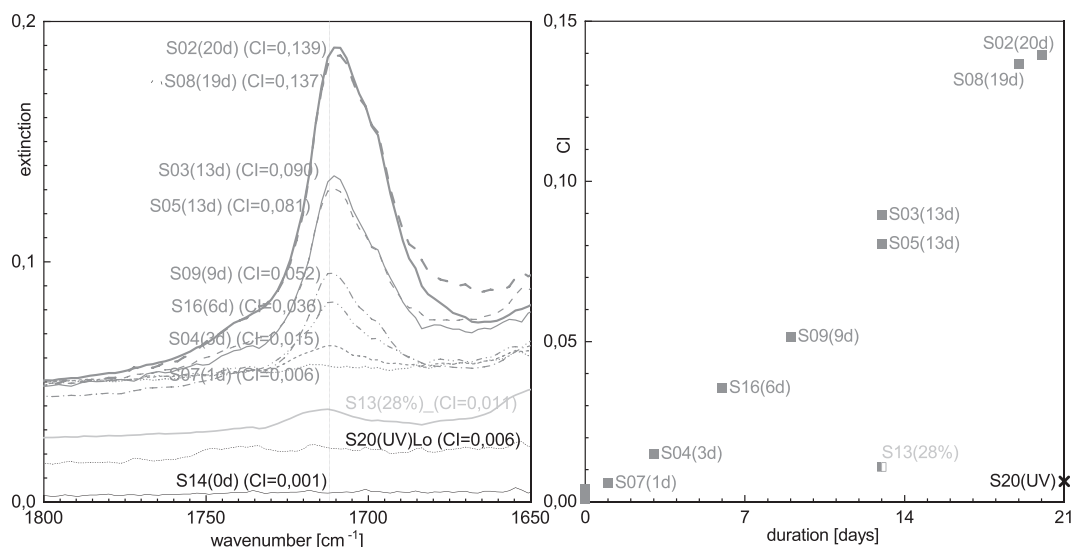
For all exposures, a decrease of the weight average  $M_w$ , compared to the original, indicates molecular degradation.

After 6 weeks of  $\text{HNO}_3$  exposure (S6), the number of extremely long chains between  $10^8$  and  $10^6\text{ g/mol}$  decreased (right from arrow 1 in Figure 7), compared to the original sample (S14). This results in a slight shift of  $M_p$  to higher masses, to which the most cleaved chains are added. No changes were noticeable in the short-chain range, revealing isolated chain breaks.

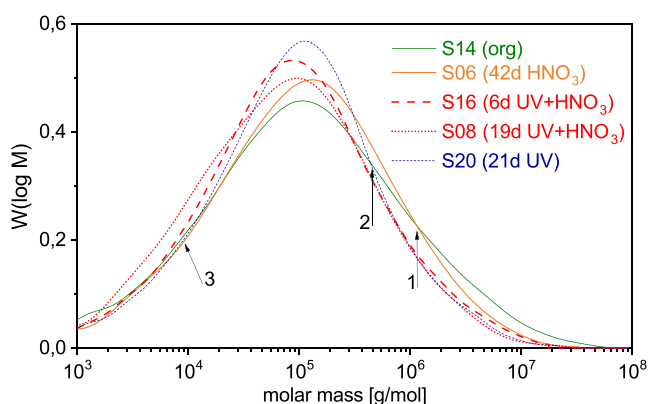
After 21 days of UV irradiation (S20), also shorter chains (down to about  $500\text{ kg/mol}$ ) were split (arrows 1 and 2 in Figure 7). The cleaved chains add up in the region around  $100\text{ kg/mol}$ , resulting in only a small shift of  $M_p$ . Here, too, no changes in the short-chain range were observed.

For the samples exposed to UV and  $\text{HNO}_3$  (S16 6d, S08 19d), even shorter chains were split, resulting in a massive increase in the short-chain fractions (arrow 3, Figure 7), increasing with exposure duration. This results in a further clear decrease of  $M_w$ .  $M_p$  shifted towards lower molar masses.

There is no indication for chain recombination or changes in branching, as claimed in Weltschev.<sup>15</sup>



**FIGURE 6** Carbonyl band formation after various exposures (left) and resulting carbonyl indices CI over exposure duration (right), with filled squares for combined UV and HNO<sub>3</sub>-55% exposure, half-filled square for combined UV and HNO<sub>3</sub>-28% exposure and cross for UV exposure



**FIGURE 7** Molar mass distribution after various exposures, normalized to the area content

**TABLE 2** Results of GPC measurement of molar mass averages for varied exposures

| Sample                         | Mn [kg/mol] | Mw [kg/mol] | Mp [kg/mol] |
|--------------------------------|-------------|-------------|-------------|
| S14 (original)                 | 17.5        | 822         | 106         |
| S6 (42d HNO <sub>3</sub> )     | 20.6        | 521         | 130         |
| S16 (6d HNO <sub>3</sub> + UV) | 19.3        | 419         | 87          |
| S8 (19d HNO <sub>3</sub> + UV) | 17.6        | 376         | 94          |
| S20 (21d UV)                   | 20.5        | 338         | 111         |

## 4 | CONCLUSIONS

A laboratory exposure device was used to simulate the damage caused to a transparent jerrycan by combined exposure to UV radiation

and the filling good. Compared to the corresponding outdoor weathering tests, the reproducibility and occupational safety have been significantly improved.

Hydraulic internal pressure tests (as component testing) as well as FTIR spectroscopy in ATR, colour measurement and molar mass investigations were used to assess the damage.

It was shown that no oxidation (FTIR) and much weaker molar mass degradation (HT-GPC) occurred after 6 weeks of pre-storage at 40°C with HNO<sub>3</sub>-55%, compared to strong oxidation after combined exposure to UV and nitric acid, which increased with the duration of exposure. This corresponds to the damage pattern in the hydraulic internal pressure test, in which the failure changes from ductile to brittle after combined exposure.

For the containers used here, this oxidation is considered critical from a duration of 6 days, which corresponds to 1/10 of an annual UV radiant exposure in Central Europe.

Furthermore, it was shown that even lower concentrations (28%) can lead to critical oxidation when the acid dissociates into nitrogen oxides, due to UV irradiation.

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## CONFLICT OF INTEREST

All authors declare that they have no conflict of interest.

## DATA AVAILABILITY STATEMENT

Data available on request from the authors.

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