Zirconia ceramics, their contrast ratio and grain size depending on sintering parameters

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This study investigated the contrast ratio and grain size depending on sintering parameters of twelve different zirconia materials and compared with glass-ceramic (N=156, n=12 per group). Contrast ratio of all ceramics was measured using a spectrophotometer according to ISO 2471: 2008. Grain sizes of zirconia were determined by SEM. Data was analyzed using one-way ANOVA followed by post-hoc Scheffe-test, Kruskal-Wallis-H-test and Spearman correlation (p<0.05). The area under the sintering curve up 25°C (AUC25) and 1200°C (AUC1200) of zirconia was calculated. Glass-ceramic showed significantly lowest contrast ratio compared to zirconia. Final sintering temperature and AUC1200 influenced contrast ratio. Grain size was affected by final sintering temperature, sintering duration and AUC. Contrast ratio and grain size showed an association.

Keywords: Zirconia, Ceramic, Contrast ratio, Grain size, Sintering parameters

INTRODUCTION

Zirconia displays among all other dental ceramics the highest mechanical properties, such as flexural strength and fracture toughness and can therefore be used for multi-unit or single tooth reconstructions even in the molar region. Clinical studies indicate that zirconia is a very suitable material for fixed dental prostheses (FDPs). A low number of fractures of zirconia frameworks have been observed while for veneered zirconia frequently chipping of the veneering ceramic occurs.

For the fabrication of zirconia in dentistry using CAD/CAM (computer aided design/computer aided manufacturing), currently two different milling procedures are available, the hard-milling of fully sintered dense blanks or the soft-milling of pre-sintered blanks, which achieve their sufficient strength by a post-machining sintering process. Milling of pre-sintered zirconia blanks is today an established process, which exhibit enough stability for milling with concurrent conservation of the milling cutters. To achieve the maximum density of the pre-sintered zirconia, the milled FDPs must additionally be sintered. Due to the material-dependent shrinkage during the sintering-procedure, the individual shrinking factor of each material-lot has to be known and considered before milling. Several studies show that despite the shrinking a very good fit of the restorations can be obtained after sintering.

Today it is possible to produce zirconia FDPs without veneering ceramic, so-called anatomic or monolithic zirconia. Translucency of such materials is essential for optimized esthetic results. Previous studies found that the translucency of zirconia can be influenced by the type and amount of additives, the sintering parameters, the heating method, and the atmospheric conditions while sintering. Particularly, the grain size, the density and the porosity of zirconia is determined by the heating method and the final sintering temperature. An increase in crystalline content in order to achieve frameworks of high strength would generally result in higher opacity. For all-ceramic reconstructions, the translucency is affected not only by the crystalline content but also by the thickness of the framework. A further study showed that the holding time during the sintering process causes grain growth in the material. The zirconia qualities of different manufacturers differ quite little in composition. Only the proportion of alumina varies according to manufacturer’s instructions in a very small range. However, there are major differences in terms of the sintering parameters such as final temperature, holding time and total sintering duration. Until now, only the influence of the single sintering parameters was examined for grain size and translucency. Hence, it is of great importance to investigate whether an interaction exists between all sintering parameters that influence the contrast ratio and grain size of zirconia ceramics.

Therefore, the aim of this study was to investigate the contrast ratio and grain size of twelve different non-colorized zirconia ceramics and to compare this with one glass-ceramic. For the evaluation of contrast ratio and grain size results, the sintering parameters are involved extensively. The tested hypotheses were that a) all tested zirconia show higher contrast ratio compared to the glass-ceramic, b) sintering parameters have an impact on contrast ratio and grain size values, and c) grain size of zirconia has an impact on contrast ratio values.
MATERIALS AND METHODS

In this study following ceramics were investigated: a) CZ: Ceramill Zi, b) VI: Vita In-Ceram YZ, c) CC: Cercon ZR, d) GC: GC ZR Disc CIP, e) PR: Prettau, f) IZ: ICE Zirkon, g) IC: InCoris ZI F0.5, h) LZ: LAVA Zirconia, i) ZE: ZENO Zr Bridge, j) CY: Copran YZ, k) DD: DD Bio Z Wiso, l) ELS: experimental laser-sintered zirconia and m) CG (glass-ceramic as control group): VITA Mark II A2. A description of all materials, manufacturers, lot numbers, and compositions can be found in Table 1.

The partially sintered zirconia and the glass-ceramic control group were cut using a low-speed diamond saw (Well 3241, Well Diamantdrahtsägen, Mannheim,

| Test group       | Abbreviation | Batch No. | Manufacturer                | Composition                                       |
|------------------|--------------|-----------|------------------------------|--------------------------------------------------|
| Ceramill Zi      | CZ           | 0904638/2 | Amann Girrbach, Koblach, Austria | ZrO₂+HfO₂+Y₂O₃ ≥99.0% Y₂O₃: 5–5.6% Al₂O₃ <0.5% other oxids <0.5% |
| Vita In-Ceram YZ | VI           | 26850     | Vita Zahnfabrik, Bad Säckingen, Germany | ZrO₂, Y₂O₃: 5% HfO₂: <3% Al₂O₃+SiO₂ <1% |
| Cercon ZR        | CC           | 20024985  | DeguDent, Hanau, Germany     | ZrO₂, Y₂O₃: 5% HfO₂: <2% Al₂O₃+SiO₂ <1% |
| GC ZR Disc CIP   | GC           | 2025      | GC Europe, Leuven, Belgium   | ZrO₂, Y₂O₃: 4.95–5.35% Al₂O₃: 0.15–0.35% SiO₂ <0.02% Fe₂O₃ <0.01% Na₂O <0.04% |
| Prettau          | PR           | ZA90002T-1| Zirkonzahn, Pustertal, Italy | ZrO₂, Y₂O₃: 4–6% Al₂O₃ <1% SiO₂: max. 0.02%, Fe₂O₃: max. 0.01%, Na₂O: max. 0.04% |
| ICE Zirkon       | IZ           | ZA90002T-2| Zirkonzahn, Pustertal, Italy | ZrO₂, Y₂O₃: 4–6% Al₂O₃ <1% SiO₂: max. 0.02 %, Fe₂O₃: max. 0.01%, Na₂O: max. 0.04% |
| InCoris ZI F0.5  | IC           | 1005300   | Sirona, Bensheim, Germany    | ZrO₂ + HfO₂ + Y₂O₃: 99% Al₂O₃ <0.5% SiO₂ <0.5% |
| LAVA Zirconia    | LZ           | 308042    | 3M ESPE, Seefeld, Germany    | Y₂O₃: 3% Al₂O₃ <0.25% ZrO₂: ~ 95% Y₂O₃: ~ 5% Al₂O₃ <1% other oxids <1% |
| ZENO Zr Bridge   | ZE           | 0810-938  | Wieland+Dental, Pforzheim, Germany | ZrO₂ + HfO₂: 94% Y₂O₃: 5% Al₂O₃ <0.4% |
| Copran YZ        | CY           | 560018    | White Peaks, Essen, Germany  | ZrO₂ + HfO₂: 94% Y₂O₃: 5% Al₂O₃ <0.4% |
| DD Bio Z Wiso    | DD           | 50389883  | Dental Direkt, Spenge, Germany | ZrO₂+HfO₂+Y₂O₃ >99% Al₂O₃ <0.25% other oxids <0.25% |
| Experimental     | ELS          | ZR060209-2| Dentaurum, Ispringen, Germany | Not available |
| laser-sintered   |              |           |                              | SiO₂-based glass ceramic |
| zirconia         |              |           |                              | |
| VITA Mark II A2  | CG           | 18090     | Vita Zahnfabrik, bad Säckingen, Germany | |

Table 1 The test groups, abbreviations, brands, batch numbers, manufacturers and composition of the tested materials.
Germany. Zirconia specimens were sintered (LHT 02/16, Nabertherm GmbH, Lilienthal/Bremen, Germany) according manufacturers’ instructions (Table 2). The experimental zirconia specimens were constructed and layer for layer lasersintered (Dentaurum). Then, all ceramic specimens were ground to the final dimensions with the thickness of 0.5±0.05 mm using SiC discs P220, P500, P1200, P2400 and P4000 (ScanDia, Hagen, Germany) in sequence. In summary, 156 specimens were fabricated. Each ceramic group included 12 specimens for contrast ratio measurements; for calculation of the correlation the same 12 specimens as for grain size analyses were used.

**Contrast ratio measurements**

The contrast ratio was measured using a spectrophotometer (CM-2600d, Konica Minolta, Hannover, Germany) according to ISO 2471: 2008 at daylight under the light source of CIE illuminant D65 brightness with color temperature of 6504 K. The measurement was made 3 times in flashing mode with an interval of 3 s, in steps of 0.1 s. Then, the software appending to the spectrometer calculated the mean values. Contrast ratios were measured from the luminous reflectance (Y) of the specimens with a black (Y_B) and a white backing (Y_W) to obtain Y_B/Y_W. In all calculations, the value “0” was considered as transparent and “1” as opaque.

**Grain size analyses**

The specimens were ultrasonically cleaned (Sonores RK102H, Bandelin electronic Berlin, Germany) for 5 min in 80% ethanol and then air-dried. Subsequently, each specimen was gold sputtered for 45 s (layer thickness: 6 nm) and surface topography was evaluated under a Scanning Electron Microscopy (SEM, Carl Zeiss Supra 50VP FESEM, Carl Zeiss, Oberkochen, Germany). SEM was operated at 5 kV and with a working distance of 5.5–7.5 mm.

**Statistical analysis**

Approximate normality of data distribution was tested using Komogorov-Smirnov and Shapiro-Wilk tests. The descriptive statistics were computed. The contrast ratio values were non-parametrically compared using Kruskal-Wallis-H test. One-way ANOVA was used for the analysis of grain size of different ceramic materials, followed by Scheffé post-hoc test. Additionally, the area under the curve (AUC) for sintering parameters was calculated. The calculation was performed for all zirconia ceramics from the room temperature (25°C/AUC25) and from the beginning of material sintering at 1200°C (AUC1200). AUC is a useful way of summarizing the information from all sintering parameters for each zirconia material separately. Straight lines to get a “curve” joined the information of heat rate, holding temperatures and times, final temperature, and cooling rate. The AUC was calculated by adding the areas under the curve between each pair of consecutive observations. Subsequently, non-parametrical correlation according Spearman was calculated between AUC25/AUC1200 and contrast ratio, AUC25/AUC1200 and grain size, sintering temperature and contrast ratio and sintering temperature and grain size of zirconia materials. P-values below 5% were considered to be statistically significant. The data were analyzed using a statistical software program (SPSS Version 21, SPSS INC, Chicago, IL, USA).

| Groups | Heat rate (°C/min) | Holding temperature and time (°C, min) | Final temperature (°C) | Holding time (min) | Cooling rate up to 25°C (°C/min) | Total sintering duration up to 25°C (min) | AUC25 (area under the curve) | AUC1200 (area under the curve) |
|--------|--------------------|----------------------------------------|------------------------|--------------------|---------------------------------|----------------------------------------|-----------------------------|-----------------------------|
| CZ     | 8                  | —                                      | 1450                   | 120                | 10                             | 440.5                                  | 399 445.3                   | 176.0                       | 37000.0                    |
| VI     | 17                 | —                                      | 1530                   | 120                | 10                             | 359.1                                  | 360 469.7                   | 172.5                       | 48262.5                    |
| CC     | 12                 | —                                      | 1350                   | 120                | 10                             | 420.0                                  | 397 500.0                   | 147.5                       | 20062.5                    |
| GC     | 12                 | —                                      | 1550                   | 120                | 12                             | 494.2                                  | 352 020.8                   | 238.0                       | 73150.0                    |
| PR     | 8                  | —                                      | 1600                   | 120                | 8                              | 513.75                                 | 499 078.1                   | 220.0                       | 68000.0                    |
| IZ     | 8                  | —                                      | 1500                   | 120                | 8                              | 488.75                                 | 448 953.1                   | 195.0                       | 47250.0                    |
| IC     | 17                 | —                                      | 1530                   | 120                | 10                             | 359.1                                  | 360 469.7                   | 172.5                       | 48262.5                    |
| LZ     | 10                 | —                                      | 1500                   | 120                | 10                             | 415.0                                  | 394 562.5                   | 180.0                       | 45000.0                    |
| ZE     | 10                 | 900, 30 further with 3°C/min            | 1450                   | 120                | 10                             | 561.5                                  | 546 298.7                   | 228.3                       | 43537.5                    |
| CY     | 6                  | —                                      | 1450                   | 120                | 10                             | 500.0                                  | 441 750.0                   | 187.0                       | 38375.0                    |
| DD     | 10                 | —                                      | 1500                   | 120                | 10                             | 415.0                                  | 394 562.5                   | 180.0                       | 45000.0                    |
RESULTS

Contrast ratio
Kolmogorov-Smirnov and Shapiro-Wilk tests indicated that 2 of 13 groups were not normally distributed. Consequently, non-parametric statistical analyses were applied. The results of the descriptive statistics (mean, SD, 95% CI) for contrast ratio measurements for each group are presented in Table 3.

The control group CG showed significantly lowest contrast ratio values of all tested groups. Within zirconia groups, the lowest contrast ratio showed LZ, PR, GC, VI and IZ. The highest contrast ratio values were observed for group CC followed by the group ELS and IC. In summary, six different significant value ranges were observed: value range A: glass-ceramic/control group (0.58±0.01); value range B: PR (0.74±0.01), LZ (0.74±0.01), GC (0.75±0.01), VI (0.76±0.01), and IZ (0.76±0.03); value range C: GC (0.75±0.01), VI (0.76±0.01), IZ (0.76±0.03), CZ (0.77±0.01), and CY (0.78±0.01); value range D: VI (0.76±0.01), IZ (0.76±0.03), CZ (0.77±0.01), CY (0.78±0.01), DD (0.78±0.02), and LZ (0.78±0.01); value range E: IC (0.81±0.01) and ELS (0.82±0.01); and value range F: ELS (0.82±0.01) and CC (0.85±0.01). Figure 1A depicts a bar diagram for the contrast ratio values. The bars of different ceramics are ordered by the increasing of contrast ratio values. Thus, left-sided of the graph the higher translucent ceramics are positioned and right-sided the more opaque ceramics.

Zirconia grain size
Kolmogorov-Smirnov and Shapiro-Wilk tests confirmed the normal distribution for all zirconia groups. Therefore, the data were compared using one-way ANOVA. The smallest grain size was observed for CC (0.07±0.01 µm), ELS (0.09±0.01 µm) and ZE (0.11±0.01 µm), respectively. In contrast, the highest grain size showed PR (0.35±0.03 µm), followed of GC (0.30±0.05 µm) and VI (0.22±0.02 µm) (Table 3). Figure 1B depicts a bar diagram for the mean grain size values. The bars of different ceramics are ordered by the decrease of grain size values from left to right.

The SEM pictures in Fig. 2 presented the zirconia grain size for each in this study tested zirconia group.

Spearman correlations
Figure 3 indicates the significantly Spearman correlations between sintering parameters (duration of sintering, final sintering temperature, and the calculated AUC25/AUC1200) and the measured contrast ratio and grain size of all twelve zirconia ceramics. Significantly negative correlations were observed between final sintering temperature and contrast ratio \( r = -0.551 \) (\( p < 0.001 \)), between AUC25 and grain size \( r = -0.400 \) (\( p < 0.001 \)), between AUC1200 and contrast ratio \( r = -0.512 \) (\( p < 0.001 \)) and between duration of sintering and grain size \( r = -0.205 \) (\( p = 0.032 \)), and

### Table 3  Mean, standard deviation (SD) values of contrast ratio and grain size with 95% confidence intervals (95% CI) of all tested groups

| Ceramics | Contrast ratio | Mean±SD | 95% CI | Mean grain size µm | Mean±SD | 95% CI |
|----------|----------------|---------|--------|--------------------|---------|--------|
| CZ       |                | 0.77±0.01<sup>CD</sup> | (0.75;0.78) | 0.12±0.01<sup>b</sup> | (0.10;0.13) |
| VI       |                | 0.76±0.01<sup>BCD</sup> | (0.73;0.77) | 0.22±0.02<sup>d</sup> | (0.20;0.24) |
| CC       |                | 0.85±0.01<sup>*</sup> | (0.82;0.86) | 0.07±0.01<sup>a</sup> | (0.05;0.07) |
| GC       |                | 0.75±0.01<sup>BC</sup> | (0.73;0.76) | 0.30±0.05<sup>e</sup> | (0.25;0.34) |
| PR       |                | 0.74±0.01<sup>IP</sup> | (0.72;0.75) | 0.35±0.03<sup>f</sup> | (0.31;0.38) |
| IZ       |                | 0.76±0.03<sup>BCD</sup> | (0.73;0.78) | 0.13±0.01<sup>b</sup> | (0.12;0.13) |
| IC       |                | 0.81±0.01<sup>IE</sup> | (0.79;0.81) | 0.21±0.02<sup>d</sup> | (0.18;0.23) |
| LZ       |                | 0.74±0.01<sup>I</sup> | (0.72;0.75) | 0.17±0.01<sup>e</sup> | (0.15;0.18) |
| ZE       |                | 0.78±0.02<sup>DO</sup> | (0.75;0.8) | 0.11±0.01<sup>ab</sup> | (0.09;0.12) |
| CY       |                | 0.78±0.01<sup>CD</sup> | (0.75;0.79) | 0.13±0.01<sup>b</sup> | (0.12;1.14) |
| DD       |                | 0.78±0.01<sup>D</sup> | (0.76;0.79) | 0.12±0.01<sup>b</sup> | (0.10;0.13) |
| ELS      |                | 0.82±0.01<sup>E</sup> | (0.80;0.83) | 0.09±0.01<sup>ab</sup> | (0.08;0.1) |
| GC       |                | 0.58±0.01<sup>A</sup> | (0.55;0.59) | —                  | —          |

<sup>* not normally distributed</sup>

<sup>ABCDEF</sup>Different superscripts represent a significant difference of contrast ratio values between all tested ceramics.

<sup>abcdef</sup>Different superscripts represent a significant differences of grain size between zirconia ceramics.
between contrast ratio and grain size \( r = -0.669 \) (\( p < 0.001 \)) as well as positive correlation between final sintering temperature and grain size \( r = 0.907 \) (\( p < 0.001 \)) and between AUC1200 and grain size \( r = 0.862 \) (\( p < 0.001 \)). In contrast, between AUC25 and contrast ratio \( r = -0.006 \) (\( p = 0.946 \)) as well as between duration of sintering and contrast ratio \( r = -0.145 \) (\( p = 0.130 \)) no correlation occurred.

**DISCUSSION**

Results obtained in this study clearly show that glass-ceramic is superior in terms of aesthetic characteristics compared to zirconia ceramics. The contrast ratio of glass-ceramic is significantly lower compared to all tested zirconia ceramics. Therefore, the first tested hypothesis, that zirconia ceramics show higher contrast ratio compared to the glass-ceramic is accepted. In this study, the contrast ratio of all specimens regardless of ceramic type was measured and compared for the same substrate thickness of 0.5 mm. It must be stressed that for glass-ceramic restorations —according to the manufacturer— the minimum thickness of 1.5 mm may not be exceeded. However, zirconia can be clinically applied with a minimum thickness of about 0.4 mm. Previous studies stated about an exponential decrease of the translucency of ceramics with an increase in thickness\(^{20,21}\). Therefore, it can be assumed that for a clinical relevant thickness of glass-ceramic the contrast ratio would increase and could be comparable with the values of the more translucent zirconia ceramics. In general, the differences concerning the clinical thickness are related to the stability of the materials. Zirconia ceramics show with approx. 1200 MPa\(^{2}\) significantly
higher flexural strength than glass-ceramic with approx. 60–150 MPa\(^2\) and can be used for clinical service with lower minimum thickness. However, conforming to the present study prior investigations showed that a higher flexural strength of ceramic resulted in lower translucency\(^2\). Additionally, variation in irradiance may be attributed to differences in crystal volume (grain size) and the refractive index. Concerning the material classes investigated in the present study, prior investigations reported for glass-ceramics a lower refractive index with 1.5\(^2\), while for zirconia ceramics a refractive index of 2.2 was measured\(^2\). Albeit not only the refractive index of the whole composition, but also the differences of the refractive indices of the single material components affect the scattering of light and therefore the contrast ratio. Less crystalline content and a refractive index close to that of the matrix causes less scattering of light\(^6\).

A previous study tested the impact of final sintering temperature of zirconia on the contrast ratio and observed that enlarged sintering temperature resulted in higher contrast ratio\(^1\). The results of the present study confirm this observation. However, in this study no correlation between contrast ratio and duration of sintering as well as the summarized sintering parameters, described as AUC25 values, could be found. This can conclude that the sintering time and heating rates play a minor role for the translucency. More important is the final temperature, which is confirmed in the calculation of the AUC from 1200°C. It can therefore be assumed that a higher amount of energy due to the higher final sintering temperature of the material is stored as a result of the longer sintering time. This in turn leads to higher amount that a grain growth takes place and thus also increase the translucency of zirconia. In contrast, grain size showed a correlation to final sintering temperature, duration of sintering and AUC measured up to room temperature at the same time. Also a negative significant correlation between grain size and contrast ratio was found. Therefore, this study showed that the complete sintering parameters affected the grain growth. On the other hand the grain size has an impact on the contrast ratio and the aesthetic properties of zirconia restorations. Hence, the second hypothesis, that the sintering parameters have an impact on

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**Fig. 3** Scatter plots for correlation between the sintering parameters and contrast ratio or grain size.

A: between contrast ratio and final sintering temperature, B: between grain size and final sintering temperature, C: between duration of sintering and grain size, D: between AUC25 and grain size, E: between AUC1200 and contrast ratio, F: AUC1200 and grain size, and G: between contrast ratio and grain size.
contrast ratio and grain size, is accepted. In general, the trend goes towards translucent zirconia. Many zirconia manufacturers tend to increase the final sintering temperature with the expectation of decreased contrast ratio. According to a previous study, the fact has to be considered that the flexural strength and stability of zirconia decrease when it was sintered above the temperature of 1550°C\(^{10}\). In this study, PR showed the highest final sintering temperature of 1600°C and the highest grain size (0.35 µm) of all tested zirconia ceramics. However, the contrast ratio results were not statistically different from the zirconia ZE, which was sintered by 1500°C and showed lower grain size (0.17 µm). Therefore, it needs to be indicated that in the rough outline the statement about the influence of sintering parameters certainly is true, but in some exceptions also additional other parameters, such as the proportion and arrangement of alumina in the zirconia matrix, have an influence on the grain size and translucency of zirconia. Numerous studies have reported that reducing the average grain size in zirconia increased the stability of the tetragonal phase\(^{15,16}\). The specimens with final sintering temperature above 1600°C produce internal stress in the zirconia surface, particularly the grains expand until the immediate neighbouring grains, which were pressed out. Consequently, this results in holes within the zirconia microstructure with the increase in grain size. Also the internal tensile stresses in the zirconia have possibly increased\(^{31,15}\). Therefore, the final sintering temperature of zirconia should not be above 1550°C.

Overall, a significant correlation was observed between contrast ratio and grain size, therefore the third hypothesis is also accepted. This is in accordance with literature data\(^{31,50}\).

At the moment no standard method exists to examine the optical properties of dental materials. Consequently it is difficult to compare the results of the present study with results of prior studies, performed on basis of other measuring methods. A prior study investigated a possible correlation between different methods and reached the conclusion that a significant correlation between most of the commonly used methods lacks\(^{26}\). In this study, the contrast ratio was evaluated using flat specimens of a standardized thickness. Future investigation should be performed directly on an anatomical restoration for greater clinical relevance. The influence of the variables related to the fabrication process for a shaded zirconia restoration, such as milling, sintering, grading and finishing could be integrated in the measurements.

A limitation of this study was the variety of different zirconia qualities. A future study should be designed using a zirconia quality, sintered with several parameters to verify these statements.

**CONCLUSIONS**

Within the limitations of this *in-vitro* study it can be summarized that the sintering parameter influenced the contrast ratio and mean grain size of zirconia. Therefore, based on the findings in this study, it can be concluded that:

1. The contrast ratio of glass-ceramic is significantly lower compared to all tested zirconia ceramics.
2. Grain size showed a correlation to the final sintering temperature, duration of sintering and AUC25/AUC1200, whereas contrast ratio only showed a correlation to the final sintering temperature.
3. A significant correlation was observed between contrast ratio and grain size as well as between contrast ratio and AUC1200.

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**Conflict of interest**

The authors report no conflict of interest. The authors alone are responsible for the content and writing of the manuscript.

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