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# New Eco-Friendly Method of Cellulosic Product Bleaching with Simultaneous Disinfection

**Abstract**
*In this article, a bleaching method with simultaneous disinfection using hydrogen peroxide in the gas phase (Vaporised Hydrogen Peroxide - VHP) is presented as an innovative, environmentally friendly, low-temperature method without wastewater generation. After the process, the cellulose products are characterised by acceptable whiteness and microbiological purity. The method presented is an eco-friendly alternative for the conventional water- and energy intensive bleaching process with reference to products made of cellulosic fibres to be applied in special applications: medical (gauze, cotton wool, bandages) and hygienic (e.g. face masks used in the prophylaxis of bacterial and/or viral infections). After tests of the sensitising effect on animals, the bleached materials were considered safe for potential users.*

**Key words:** *vaporised hydrogen peroxide, cotton bleaching, disinfection, microbiological purity.*

## Introduction

Cotton cellulose/cotton fibres contain a significant level of impurities such as pectin (0.4 - 1.2%), waxes (0.4 - 1.2%), protein (1.0 - 1.9%), inorganic salts (0.7 - 1.6%) and others – resins, pigments, and hemi-cellulose (0.5 - 0.8%) [1].

Non-cellulose substances, rendering the hydrophobic character, are removed during the pre-treatment processes. Despite this, cellulose fibre still has yellowish or brown coloration derived from organic substances permanently bound with fibres. The elimination of this colour may occur by changing the system of electrons in chromopfores of natural dye molecules occurring in cellulosic fibres in the bleaching process using oxidising agents.

Bleaching methods once used were based on the use of chlorine compounds. Despite the high oxidation potential of these, the bleaching capabilities at ambient temperature and the relatively low price, they were withdrawn due to adsorbed, organic chlorine compounds generated in these processes (AOX). Alternative, oxidising bleaching agents are hydrogen peroxide and peracetic acid [2 - 9]. The bleaching of products made of cellulose fibers - flax and cotton, using peracetic acid (10 - 15%) allows to obtain water (the bleaching process is carried out at pH 6 - 8, which enables to reduce the amount of rinsing operations after bleaching) and energy savings (lower temperature of bleaching 60 - 80 °C) and the bleaching time is shortened [2, 3]. Nowadays, due to the environmental approach, the most prevalent in textile industry is the bleaching of textiles made of cellulose fibers with hydrogen peroxide. Bleaching is carried out at a high temperature for a relatively long time in strongly alkaline media (pH > 11) and it requires the use of stabilisers. After the bleaching process, it is necessary to remove from the bath stabilizers and residues of hydrogen peroxide, which may cause the chemical degradation of cellulose. Therefore energy and water consumption in this process and during the preceding pre-treatment is very high, with an average of 100 - 150 $dm^3$ of water per 1 kg and from 5 to 20 kWh of energy per 1 kg of the textile product [10].

Environmental care and the need to meet increasingly stringent standards for water and wastewater still force to seek new technological solutions to eliminate impurities, preferably through the use of waste-free methods. The use of gas phase hydrogen peroxide in bleaching is certainly an innovative solution. In the literature there is no reference to such a method of bleaching textile products. It is known, however, that hydrogen peroxide in the gas phase (Vaporised Hydrogen Peroxide - VHP) has been previously used in (bio) the pharmaceutical industry to disinfect the surfaces of production lines and facilities, as well as in health care centers for disinfection of the surfaces of medical instruments, diagnostic equipment, hospital and laboratory rooms [11 - 15]. Hydrogen peroxide in the gas phase operates on a plurality of microorganisms, both in vegetative - Gram-positive and Gram-negative bacteria, fungi, viruses, and as a spore, which is considered to be the most resistant to the various cidal agents [15 - 17]. Hydroxyl radicals generated by the decomposition of hydrogen peroxide in a gas form oxidize elements of the structure of microbial cells (cell membrane lipids, DNA), leading to their destruction [12]. Vaporised hydrogen peroxide [VHP] shows more efficient antibacterial activity than hydrogen peroxide in a liquid form. As an example, VHP at a concentration of 1 $mg/dm^3$ is active towards bacterial culture atabout 400 $mg/dm^3$ $H_2O_2$ in a liquid form [18].

The bleaching of textile materials using vaporised hydrogen peroxide is not a well known process described in scientific literature. Available publications refer only to bleaching with $H_2O_2$ in the liquid phase (water baths). Bleaching textile materials using VHP is a multi-step process consisting of the following physical-chemical processes:

- $H_2O_2$ diffusion to the surface of the textile material,
- Adsorption process,
- Reaction of $H_2O_2$ with a colour contaminant contained in the cotton fiber,
- Desorption of gaseous reaction products (oxygen, water vapor) and unreacted hydrogen peroxide,
- $H_2O_2$ decomposition reaction in the gas phase,
- Water vapour sorption.

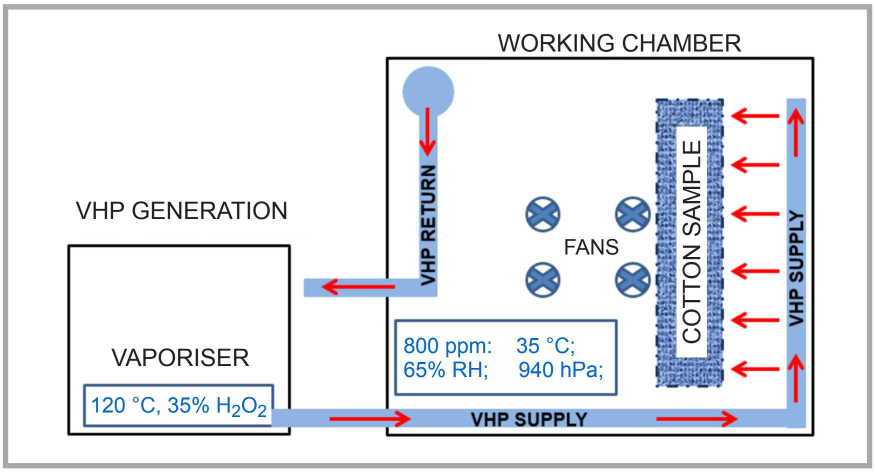


***Figure 1.** Schematic diagram of VHP generation system.*

In the description of the bleaching process in the liquid phase, it is assumed that the rate of the total process is limited by the chemical reaction speed. It appears that the gas-phase process is also controlled by a chemical reaction.

Hydrogen peroxide in the gas phase is produced by the vaporisation of liquid hydrogen peroxide (at the concentration of 30 to 59%) at 120 °C, whereby the mixture of VHP and water vapor is obtained. Presented in this paper is a treatment process for textiles made of cellulosic fibers with VHP, which is a dry process wherein the VHP concentration is maintained below the condensation point. The value of this point depends on the ambient temperature. If the VHP concentration exceeds the saturation point at a given temperature, it is followed by the condensation of concentrated hydrogen peroxide on surfaces, due to differences in evaporation pressures of the hydrogen peroxide solution - $P_{VHP}$ and water - $P_{H_2O}$ ($P_{VHP} < P_{H_2O}$). In the VHP process, condensation is an undesirable phenomenonbecause the process takes place in an uncontrolled manner and is potentially dangerous to both the surfaces of the textile product (because of the possibility of damage) and the measuring instruments used for monitoring conditions within the chamber (due to the possibility of corrosion) [19, 20].

The VHP treatment process is a fully ecological process, as a result, no wastewater is generated, and by-products are decomposition products of hydrogen peroxide: water and oxygen.

In this study, cotton gauze was subjected to VHP treatmen,t and then its properties such as whiteness, the polymerisation degree, and mechanical and some hygienic properties were examined. It was also an objective of this work to investigate results of the decontamination efficiency of cotton gauze contaminated with selected strains of microorganisms.

## Experimental

### Materials

Experiments were carried out for unbleached 100% cotton gauze of 17 threads per 1 $cm^2$ - 10 in the warp direction and 7 in the weft direction (TZMO, Poland), with area mass 28 $g/m^2$. Before testing, cotton gauze samples were subjected to pre-treatment with an anionic wetting-washing agent - Periwet WLV (Dr. Petry GmbH, Germany) - 2 g/l.98 °C, 30 min.

### *Chemical agents*

- Hydrogen peroxide 35% (Nitrogen Company, Puławy, Poland),
- Sodium hydroxide.

### *Microorganisms*

- Bacteria strains: *Staphylococcus aureus* ATCC 6538,
- Bacterial spores: *Bacillus subtilis* (BGA, by Merck) ATCC 19659, *Bacillus atrophaeus* ATCC 9372 (Simicon GmbH)
- Fungi: *Aspergillus niger* van Tieghem ATCC 6275,
- Fungi spores: *Chaetomium globosum* Kunze: Fries ATCC 6205

### *Nutrient mediums*

- Tryptone soya agar (Difco$^{TM}$)
- Tryptone soya broth (BTL)
- Czapek-Dox agar (BTL)
- Czapek-Dox broth (BTL)

### Bleaching and decontamination process with VHP

Samples were subjected to bleaching with decontamination treatment with the VHP process in a prototype VHP device (product of Innovation-Development Enterprise *IMPULS*, Poland), shown in ***Figure 1***, under controlled conditions: recommended mean VHP concentration of 800 ppm, temperature of 35 ± 2 °C, relative humidity of 65 ± 4%, pressure of 940 hPa, and times of 1, 2, 4 or 8 hours. On the basis of earlier studies it was found that the preferred time of VHP treatment is 4 hours. VHP treatment of cotton textiles is a periodic process consisting of the following phases (***Figure 2)***:

- Saturation of the working chamber with VHP to assumed concentration (e.g. 800 ppm);
- Bleaching/decontamination – in this phase the VHP concentration is maintained at the defined level by making it up,
- Aeration – supply of VHP to the working chamber is suspended while the system carries away the mixture of VHP and steam until a safe concentration (in a closed cycle) is obtained.

For comparison, the traditional peroxide bleaching process of cotton gauze in a bath containing 4 $g/dm^3$ of 35% $H_2O_2$, 2 $g/dm^3$ of NaOH and auxiliaries was carried out at atemperature of 98 °C for 60 minutes. The lquor ratio amounted to 1:10. After bleaching there was rinsing at a temperature of 80 °C and 60 °C, then cold rinsing and rinsing with 1 $g/dm^3$ of 50% acetic acid.

### Whiteness measurement

Bleaching results were evaluated by measuring the degree of whiteness for cotton gauze samples - *CIE whiteness*

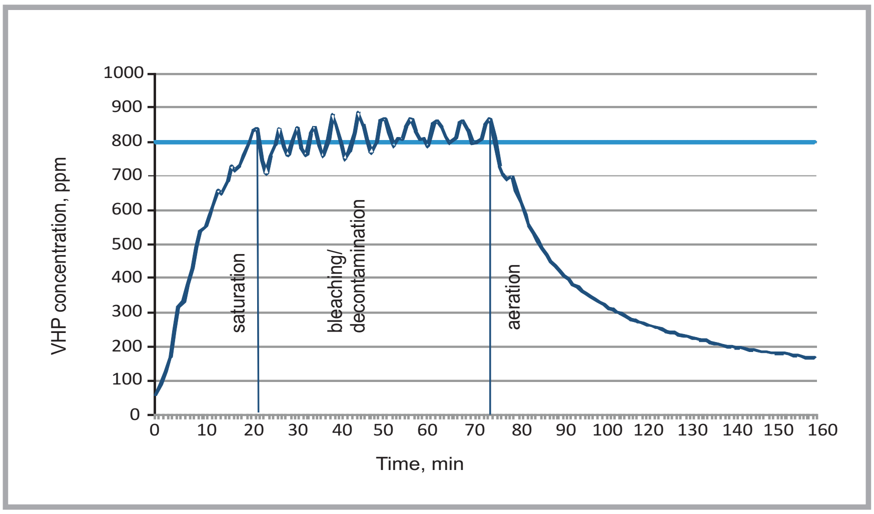


***Figure 2.** Cycle of bleaching/decontamination with the use of VHP.*

*index* (WI) with a Datacolor 650 (Datacolor Int.) spectrophotometer equipped with Datacolor Tools software according to EN-ISO 105-J02 for a 10° observer, by $D_{65}$ light. The colorimetric coordinates (X, Y, Z) of the gauze samples and the chromaticity coordinates (x, y) were calculated. Then the whiteness index was determined according to CIE formula (1)

$$WI_{CIE} = Y + 800(x_0 - x) + 1700(y_0 - y) \quad (1)$$

where, $x_0$ and $y_0$ are the coordinates of the achromatic point for the given illuminant. For the $D_{65}$ and 10° observer these values amount to 0.3138 and 0.3310, respectively. This CIE whiteness index formula is limited to the value 40 < WI < 5Y – 280.

### Degree of polymerisation

The polymerisation degree was determined by the viscosimetric method given in ISO 5351 – *Determination of the limiting viscosity number in cupriethylenediamine (CED) solution*. This method relies on the determination of the viscosity ratio of cellulose polymer solution in cupriethylenediamine (CED) by measuring the efflux time of the solution.

### Mechanical properties

The tensile strength was determined according to EN ISO 13934-1 with the use of a tensile testing machine - Hounsfield H5KS (Tinius, Ldt, England), with a velocity of tension of 100 m/min and distance between clamps of 200 mm.

### Hygienic parameters

The pH value of water extracts for the cotton gauze samples was determined according to EN-ISO 3071, with the following type of extraction solution: deionised water with pH 5.2, temperature 22.1°C. The water absorption ratio was determined according to Polish standard PN-89/P-04781/12.

Cotton gauze samples – 5 g each, were placed in a metal basket and immersed in distilled water for 2 minutes at ambient temperature. Then the basket with a sample was taken out, the excessive water drained off gravitationally for 30 sec, and then it was weighed. Water absorption ratio A for each sample was calculated according to formula (2):

$$A = m_{ws} / m_{ds} \quad (2)$$

where, $m_{ws}$ – mass of wet gauze sample after immersion in g, $m_{ds}$ – mass of dry gauze sample before immersion in g.

### Microbiological tests procedure

Selected bacteria strains: *S. aureus* ATCC 6538, bacterial spores *B. subtilis* ATCC 19659 and *B. atrophaeus* ATCC 9372 as well as fungi: *A. niger* ATCC 6275 and *Ch. globosum* ATCC 6205 were deposited on the cotton gauze samples, with an amount of suspension of 0.1 ml per sample. Then the contaminated gauze samples were subjected to treatment with VHP processes: at 200 ppm or 500 ppm of VHP at 35 °C for 20 minutes. After the VHP process, the samples were placed on plates with appropriate nutrient agar (*Tryptone soya agar* by Difco™ for bacteria and spores; *Czapek-Dox agar* by BTL for fungi) or into an appropriate broth (*Tryptone soya broth* by BTL for bacteria and spores, *Czapek-Dox broth* by BTL for fungi). Then these samples as well as control ones (gauze samples contaminated with individual microorganisms) were put into a laboratory incubator in conditions optimal for individual microorganism growth: temperature of 37 °C, time -72 hours for bacteria, temperature of 30 °C, time - 168 hours for spores and temperature of 30 °C, and time - 192 hours for fungi.

Gauze samples were considered as sterile if during the incubation period there was no microbial growth on the plates with agar nor in the broth.

### Tests of sensitizing effect on animals

Tests *in vivo* for animal – guinea pig skin sensitization for samples of cotton gauze of treated by vaporised hydrogen peroxide were performed in accordance with OECD guidelines by the Research Laboratory of Medicine and Veterinary Products using the GMP Quality System at the Nofer Institute of Occupational Medicine in Lodz, Poland.

## Results and discussion

The use of hydrogen peroxide in the gas phase in the treatment of gauze caused a significant increase in the whiteness degree of this fabric (***Table 1***) from 3.27 for an unbleached sample to 55.13 for a sample treated at the recommended VHP concentration of 800 ppm for 8 h. The whiteness degree of VHP treated cotton gauze increased with the length of the process. Prolonged treatment with VHP at an 800 ppm concentration from 1 to 8 hours resulted in an increase in the whiteness degree of cotton gauze from *WI* = 43.09 to *WI* = 55.13. The whiteness degree of cotton gauze obtained by treatment with VHP (*WI* = 55.13 at 8 hrs of treatment) is lower than for the bleached with hydrogen peroxide in a bath (*WI* = 74.23), and for a commercial compress of gauze (*WI* = 66.01). However, the characteristics / technical requirements for medical products, such as gauze dressings, do not specify a minimum degree of whiteness which should characterise this type of product. After organoleptic evaluation it was found that samples of VHP bleached gauze were very pleasing to the eye and had an aesthetically acceptable shade of white.

It is known that the degree of polymerization of cellulose may be reduced by reaction with hydrogen peroxide [21]. In the case of applying both methods of bleaching, using gas and liquid $H_2O_2$, a reduction in the degree of polymerisation

*Table 1. Degree of CIE whiteness index for cotton gauze fabric after the bleaching process.*

| Sample of cotton gauze | | CIE Whiteness index |
|---|---|---|
| Unbleached | | 3.27 |
| Bleached with VHP (800 ppm, 35 °C, RH 65%) | 1 h | 43.09 |
| | 2 h | 46.75 |
| | 4 h | 51.50 |
| | 8 h | 55.13 |
| Bleached in bath with $H_2O_2$ (4 g/l of 35% $H_2O_2$, 2 g/l NaOH and auxiliary agents, 98 °C, 1 h, liquor ratio 1:10) | | 74.23 |
| Commercial 17-thread gauze compress | | 66.01 |

*Table 2. Polymerisation degree and tensile strength for cotton gauze fabric after the bleaching process.*

| Sample of cotton gauze | Polymerisation degree DP | Tensile strength, N |
|---|---|---|
| Unbleached | 3227 | Warp 65 ± 1.0<br>Weft 54 ± 3.7 |
| Bleached with VHP<br><br>(800 ppm, 35 °C, RH 65%, 4 h) | 1150 | Warp 62 ± 5.1<br>Weft 50 ± 1.7 |
| Bleached in bath with $H_2O_2$ (4 g/l of 35% $H_2O_2$, 2 g/l NaOH and auxiliary agents, 98 °C, 1 h, liquor ratio 1:10) | 2566 | Warp 66 ± 3.6<br>Weft 53 ± 3.9 |

*Table 3. pH values and water absorption ratio for cotton gauze fabric after the bleaching process.*

| Sample of cotton gauze | pH | Water absorption ratio |
|---|---|---|
| Unbleached | 6.7 | Does not absorb |
| Bleached with VHP<br>(800 ppm, 35 °C, RH 65%, 4 h) | 6.6 | 12.0 |
| Bleached in a bath with $H_2O_2$ (4 g/l of 35% $H_2O_2$, 2 g/l NaOH and auxiliary agents, 98 °C, 1 h, liquor ratio 1:10) | 6.5 | 11.6 |

was noticed compared to the unbleached sample (***Table 2***). A clearly observed change in the degree of cellulose polymerisation by $H_2O_2$ action affected the change in the mechanical properties of the bleached textile materials to a small extent (***Table 2***). The average value of the tensile strength - for the unbleached sample 65 N in the warp direction and 54 N in the weft direction - practically did not change after the bleaching process of cotton gauze in a bath containing $H_2O_2$ and amounted to 66 N and 53 N, respectively. However, after the VHP process a slight decrease was observed in the tensile strength, which in this case amounted to 62 N and 50 N, respectively. On the basis of earlier studies, it was found that only a decrease in the polymerization degree below 500 caused a significant reduction in the tensile strength value.

For products for medical applications, which include gauze dressings, hygienic parameters are very important. According to the generally accepted requirements, the pH of the aqueous extract from such products should be inert to the human skin and equal from 6.5 to 7.5. As is apparent from the results shown in ***Table 3***, the pH value of the various gauze decreased slightly as a result of the two bleaching processes, both in a bath containing $H_2O_2$ - the pH was lowered from 6.7 to 6.5, and after VHP bleaching - the pH was lowered from 6.7 to 6.6. Even so, the pH of the cotton gauze treated with both gaseous and liquid hydrogen peroxide is within the range ensuring the safety of the human skin.

Another important parameter of the gauze hygienic characteristic is its absorption ability. The absorbency determined for gauze samples treated with gaseous and liquid hydrogen peroxide was 12 and 11.6, respectively (***Table 3***). Thus as a result of the VHP bleaching of cotton gauze, similar (slightly higher) sorption properties were obtained as for the conventional bleaching process.

Studies of microbiological purity based on selected reference strains of bacteria, bacterial spores and fungi were implemented using two types of microbiological media - agar and liquid medium, used in order to provide better penetration into the gauze samples tested. Research results show (***Table 4***) that VHP treatment at relatively low concentration (200 ppm) resulted in effective disinfection of cotton gauze samples infected by the following strains of bacteria: *Staphylococcus aureus*, *Bacillus subtilis* and spores *Bacillus atrophaeus,* and fungus *Chaetomium globosum*.

In the case of gauze contaminated by the fungus *Aspergillus niger*, and then incubated on a liquid medium, the effi-

*Table 4. Decontamination efficiency of cotton gauze samples contaminated with selected microorganisms strains by means of VHP.*

| Microorganism | Density of bacterial suspension deposited on gauze samples, CFU/ml | Condition of VHP treatment | Nutrient medium | Broth of the microorganism on the sample | |
|---|---|---|---|---|---|
| | | | | Control - without decontamination | After decontamination with VHP |
| *Staphylococcus aureus*<br>ATCC 6538 | $1.4x10^8$ | VHP concentration 200 ppm, temperature 35°C, time 20 minutes | *Tryptone soya agar* | growth | no growth |
| | | | *Tryptone soya broth* | | |
| Spores of *Bacillus subtilis*<br>ATCC 19659 | $1.2 x10^7$ | | *Tryptone soya agar* | | |
| | | | *Tryptone soya broth* | | |
| Spores of *Bacillus atrophaeus*<br>ATCC 9372 | $1.9x10^7$ | | *Tryptone soya agar* | | |
| | | | *Tryptone soya broth* | | |
| *Chaetomium globosum*<br>ATCC 6205 | $0.2 x10^6$ | | *Czapek- Dox agar* | | |
| | | | *Czapek- Dox broth* | | |
| *Aspergillus niger*<br>ATCC 6275 | $7.4 x10^6$ | | *Czapek- Dox agar* | | |
| | | | *Czapek- Dox broth* | | growth |
| *Aspergillus niger*<br>ATCC 6275 | $7.1 x10^6$ | VHP concentration 500 ppm, temperature 35 °C, time 20 minutes | *Czapek- Dox agar* | | no growth |
| | | | *Czapek- Dox broth* | | |

ciency of VHP at a concentration of 200 ppm proved to be insufficient. Treatment with a higher VHP concentration - 500 ppm, without changing the other process conditions (temperature and treatment time), allowed efficient sterilization of cotton gauze infected by the *Aspergillus niger* strain. Studies of microbiological purity of the infected cotton gauze with various microorganisms were carried out under much milder conditions (i.e. relatively low VHP concentration of 200 or 500 ppm, short processing time - 20 minutes) than the recommended conditions for the bleaching process (800 ppm, 4 hours). Therefore the use of the VHP treatment of cotton gauze under conditions necessary to obtain the highest possible whiteness allows to sterilize cotton gauze with reference to all microorganisms considered in this article: *Staphylococcus aureus, Bacillus subtilis* and spores *Bacillus atrophaeus globusom,* and fungi *Chaetomium and Aspergillus niger*.

The results of studies of animal skin sensitization confirmed that cotton gauze subjected to a bleaching process with simultaneous disinfection using vaporized hydrogen peroxide is a safe product for potential users.

## Conclusions

Treatment of cotton gauze with VHP under recommended conditions: VHP concentration 800 ppm, temperature 35 °C, process time - 4 hours, allows to obtain bleached textile products characterised by an acceptable whiteness and microbiological purity - devoid of the following microorganisms: bacteria - *Staphylococcus aureus,* spores *Bacillus subtilis* and *Bacillus atrophaeus,* as well as fungi - *Chaetomium globusom* and *Aspergillus niger*.

It was found that the bleaching process takes place within the first hour and its rate depends on the speed of the chemical reaction on the fiber surface.

The process utilizing hydrogen peroxide in the vapour phase virtually has no influence on the change of mechanical properties (tensile strength). As a result of VHP bleaching good hygienic performance of cotton gauze was achieved, similar to the result for bleaching with hydrogen peroxide in a bath.

The bleaching process with simultaneous disinfection using vaporised hydrogen peroxide is environmentally friendly, at low-temperature, energy-saving and waste-free, and as such is an alternative to the conventional water-and energy-consuming bleaching process using aqueous hydrogen peroxide for products for special applications: medical (gauze, cotton wool, bandages), and hygienic - protective masks for the human respiratory system used in the prophylaxis of bacterial and/or viral disease transmission.

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