

A Comprehensive Multimethod Analysis of Mechanical Properties of two different heat treatments for endodontic Nickel-titanium instruments

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Abstract

The purpose of the present study was to evaluate the influence of the two different heat treatments of the mechanical properties of two commercial products: X7 Firewire and X7 Utopia Nickel-titanium rotary instruments (EdgeEndo, Albuquerque, New Mexico). The present article was written following the guidelines of PRILE (Preferred Reporting Items for Laboratory studies in Endodontology). Since both instruments are available with same design, size and taper the influence of the two different heat treatments on various mechanical properties could be properly evaluated. 50 instruments for each group, as determined by power analysis, were selected and tested using methods and devices validated in previous studies. Data were collected and statistically analyzed using a 1-way ANOVA test followed by the post hoc Tukey test with significance set to a 95% confidence level. Results showed that X7 Utopia were found to be more rigid, with a statistically significant difference when compared to X7 Firewire. Similarly, X7 Utopia were found to be significantly more resistant to torsion, while no statistically significant difference was found between the two tested instruments when subjected to a cyclic fatigue test. Cutting efficiency was significantly higher for X7 Utopia instruments, and also instrumentation time was significantly shorter when compared with X7 Firewire. X7 Firewire instruments showed a higher tendency to flutes deformations during usage. We can conclude that thermal treatments of nickel-titanium instruments can significantly impact all their mechanical properties in vitro and overall performance in root canal procedures. Therefore, understanding the differences in thermal treatments is crucial for manufacturers to improve instruments and for dental professionals to tailor these instruments to specific clinical requirements.

Key words: endodontic instruments, nickel-titanium, heat treatment

Introduction

Nickel-titanium (NiTi) instruments are widely used in endodontics due to their flexibility, shape memory, and resistance to cyclic fatigue. In the last decades NiTi rotary instrumentation technique have been considered as golden standard to achieve proper canal shaping with a more efficient, rapid and simple clinical approach, even if intracanal separation or rotary instruments is still a major concern for the majority of clinicians (1). In the last decades improvements in design, motions and manufacturing have been proposed to provide clinicians with safer and more efficient NiTi rotary instruments. In recent years, however, the majority of manufacturers have focused their interest in improving heat treatment (HT) procedures before, during or after the grinding process (1,2). Several studies have shown the benefits of such treatments; however,

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they are proprietary and not disclosed by manufacturers (3-5). It has been shown that many mechanical properties can be influenced by the heat treatments, even if overall performance is still a combination of instruments' design, dimensions, motions and alloy (6-8). Currently the heat treatment of endodontic NiTi instruments has become a crucial aspect of their manufacturing process, playing a pivotal role in enhancing their mechanical properties and overall performance in root canal procedures. Heat treatment involves subjecting the NiTi alloy to specific temperature and time conditions to modify its microstructure. The primary goals of heat treatment for endodontic NiTi instruments are to enhance their flexibility, resistance to fracture, and to establish a balanced combination of hardness and toughness (5-8).

The process typically involves a sequence of steps, including solution treatment, quenching, and aging. During solution treatment, the NiTi alloy is heated to a temperature where it transitions from a martensitic to an austenitic phase. This phase transition is critical for imparting the desired shape memory and superelasticity to the instruments. Quenching follows, where the heated alloy is rapidly cooled to lock in the austenitic phase and achieve the desired mechanical properties. Subsequently, aging is performed to optimize the balance between hardness and toughness, ensuring the instrument's durability during clinical use (1,7). The controlled application of heat treatment mainly addresses some of the challenges associated with NiTi instruments, such as their susceptibility to cyclic fatigue and potential for breakage. The process enhances the instruments' fatigue resistance by refining the grain structure and controlling phase transformations, leading to a more robust and reliable endodontic tool (9). Manufacturers continually refine heat treatment processes to tailor NiTi instruments for specific clinical applications (10).

X7 instruments are manufactured by Edge Endo (Albu-

querque, New Mexico, USA) and have a constant taper, a parabolic cross-section, a non cutting tip and a 1mm maximum flute diameter. Even if design and dimensions are same the X7 NiTi rotary instruments are currently commercialized with two different names, due to two different types of heat treatment. "Fire-Wire" X7 instruments are made of an Annealed Heat Treated (AHT) nickel-titanium alloy brand named Fire-Wire (more ductile). According to the manufacturer (11) such alloy improves flexibility, resistance to cyclic fatigue and reduces bounce-back effect inside curvatures, and instruments closely follow the anatomy of the canal without straightening out, reducing the risk of ledging, transportation, and perforation. It also allows the NiTi rotary instruments to be easily straightened with clinicians fingers (prebendable). The EdgeX7 "Utopia" is a more recently commercialized, NiTi rotary instrument that, according to manufacturer, provides all of the benefits of the original Firewire X7 blade design while taking performance to a different level with more cutting efficiency, due to a different proprietary heat -treatment (11).

Since both instruments are available with same design, sizes and tapers, and are meant to be used with the same protocol and same motion's parameters (rotational speed and torque), the purpose of the present study was to evaluate the influence of the two different heat treatments of the mechanical properties of the two commercial products (X7 Firewire and X7 Utopia instruments). The null hypothesis was that no changes in the in vitro mechanical properties were provided by the different heat treatments.

Materials and Methods

The present article was written following the guidelines of PRILE (Preferred Reporting Items for Laboratory studies in Endodontology (Nagendrabu 2021) , as shown in the flow chart of the study (Fig.1).

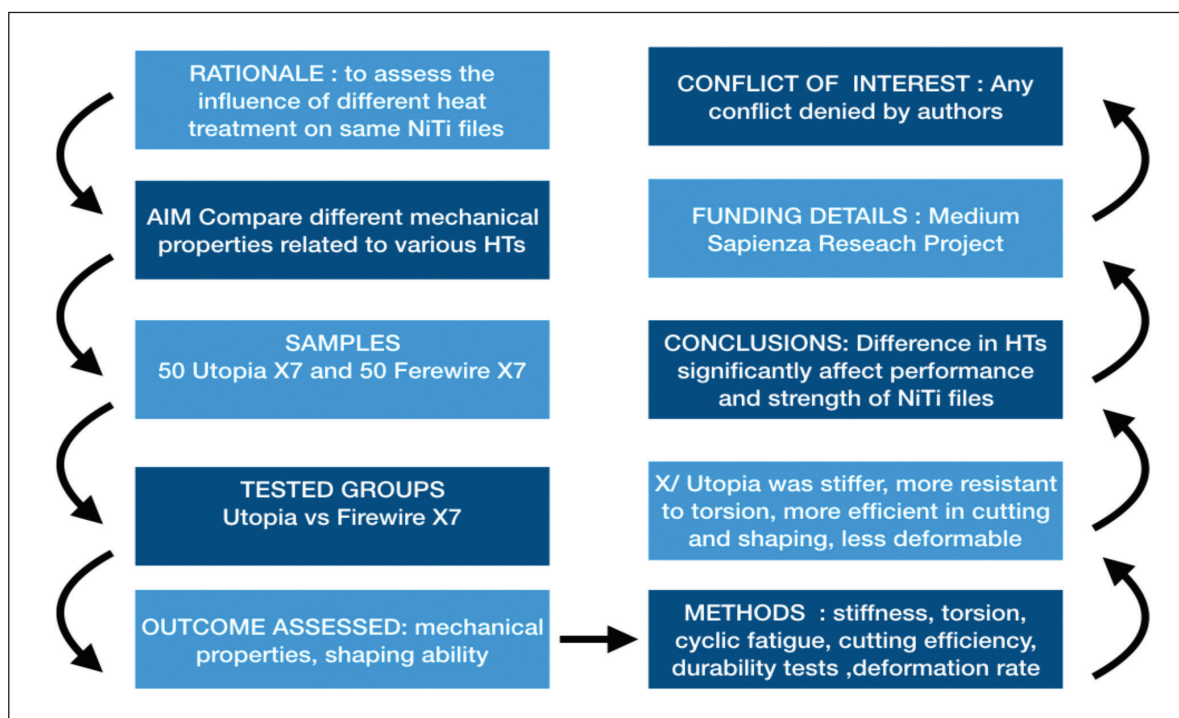


Figure 1.

Sample selection

A total of 50 new X7 NiTi instruments and 50 new XT Utopia NiTi instruments size 25 and .04 taper (EdgeEndo, Albuquerque, New Mexico, USA) were selected for the study and divided in 5 groups of ten each (Fig 2a). Each group was then subjected to one of the following mechanical tests: stiffness, cyclic fatigue, torsional resistance, cutting efficiency, durability. According to manufacturer all the instruments have same design and dimensions, while they differ only due to different heat treatments, even if such manufacturing processes are proprietary and not disclosed (11).

Before starting the laboratory assessment all the 100 instruments were examined under dental stereomicroscope (Kaps, Asslar, Germany) at x10 magnification to identify major irregularities or defects in the blade design, which could affect properties of the NiTi instruments and make them not valid for the investigation. No instrument was discarded and all 100 samples were accepted for the study.

Sample size for each mechanical test was determined by power analysis and calculated based on preliminary data obtained after 6 initial measurements with a power of 80% and a 0,05 alpha type of error. For the five previously mentioned tests sample size calculations were 3, 4, 3, 4 and 6 respectively and, consequently, a total number of 10 instruments per group was considered more than enough for each dependent variable.

Stiffness Test

Ten instruments for each product underwent the stiffness test (resistance to bending stress). The stiffness tests were performed using a device, which has been used in previously published peer-review studies (12) and follows ISO 3630-1 international standard guidelines for mechanical tests of endodontic instruments (fig 2b). The device consists of a load cell, an electronic display, and a mobile holder to allow repeatable positioning of the instruments on the load cell. The stiffness tests were performed by bending each file at a 45° angle at 3 mm from its tip and recording the applied force (g). The measurements indicated by the electronic display connected to the load cell were recorded. The higher the values, the stiffer (less flexible) the instrument was. Mean values, the standard deviations and statistical significance of the cyclic fatigue tests are displayed in table 1.

Cyclic fatigue test

Ten instruments for each product were subjected to the test. All instruments were rotated in a stainless-steel artificial canal of 16 mm characterized by a 90° angle of curvature and a 2-mm radius of curvature (fig 2c) using glycerin as a lubricant to avoid any friction between the files and the artificial canal. The methodology has been validated by many studies published in peer-review indexed journals (13-15). Speed (300 rpm clockwise) and torque (2N) were selected according to the manufacturers' recommendation and each test was performed by the same expert operator. Each instrument was carefully inserted at the same length (16mm) and rotated inside the canal until a visible and/or audible sign of fracture was detected. The time to fracture (TtF) was measured using a digital chronometer with a sensitivity of 0.01 seconds. The test was performed at room temperature. The

length of the fragments (FL) was measured with a digital caliber and statistically analyzed to evaluate the correct positioning of the instruments inside the artificial canal and to verify the comparability of the results of the cyclic fatigue test. All data were statistically analyzed using a 1-way ANOVA test followed by the post hoc Tukey test with significance set to a 95% confidence level. Mean values, the standard deviations and statistical significance of the cyclic fatigue tests are displayed in table 1.

Torsional test

Ten instruments for each product underwent the torsional resistance test using a methodology which has been validated by studies published in peer-review indexed journals (16) and follows ISO Guidelines 3630-1. Tests were performed with a custom-made torsionmeter-like device at 300 rpm, because it has been demonstrated that rotational speed does not affect the results. The device allowed to avoid the bending of the coronal part of the instrument and to have a straight angle of insertion, since it has been demonstrated that such coronal interferences and stresses can deeply influence the torsional resistance. The test was performed blocking the tip of the instrument with a vise at 3 mm from the tip (fig 2d) and rotating it at 300 rpm in the clockwise direction with a dedicated electronic motor (Kavo, Biberach, Germany) allowing a real-time (0.1 seconds) recording of the torque with a sensitivity of 0.05 Ncm. The torque at fracture results were collected on a spreadsheet. The length of the fragments (FL) was measured with a digital caliber and statistically analyzed to evaluate the correct positioning of the instruments' tip inside the torsionmeter and to verify the comparability of the results of the torsional test.

All data were statistically analyzed using a 1-way ANOVA test followed by the post hoc Tukey test with significance set to a 95% confidence level. Mean values, the standard deviations and statistical significance of the torsional tests are displayed in table 1

Cutting efficiency test

Ten instruments for each product underwent the cutting efficiency test using a methodology (Fig 2e) which has been validated by studies published (17,18) in peer-review indexed journals. The device consisted of a main frame to which a mobile plastic support for the handpiece was connected and a stainless-steel block containing the Plexiglas plates (Inplex, Rome, Italy), against which the cutting efficiency of the instruments was tested. A notch 1 mm in depth and width had been created on the lateral wall of the Plexiglas plate that measured 1 mm in thickness, to prevent the instruments from slipping out the smooth surface of the plastic. The dental handpiece was mounted upon a mobile device connected to a fixed weight (150 g), that for gravity drove the horizontal instrument against the Plexiglas block in a precise and reproducible way. The plastic support for the handpiece allowed for precise and simple three-dimensional alignment and positioning of the instrument, as soon as it came perpendicularly into contact with the notch created on the wall of the Plexiglas specimen without bending. Once everything was fixed, the motor of the testing device was switched on and the instrument removed material and penetrated actively. The cutting efficiency was tested 6

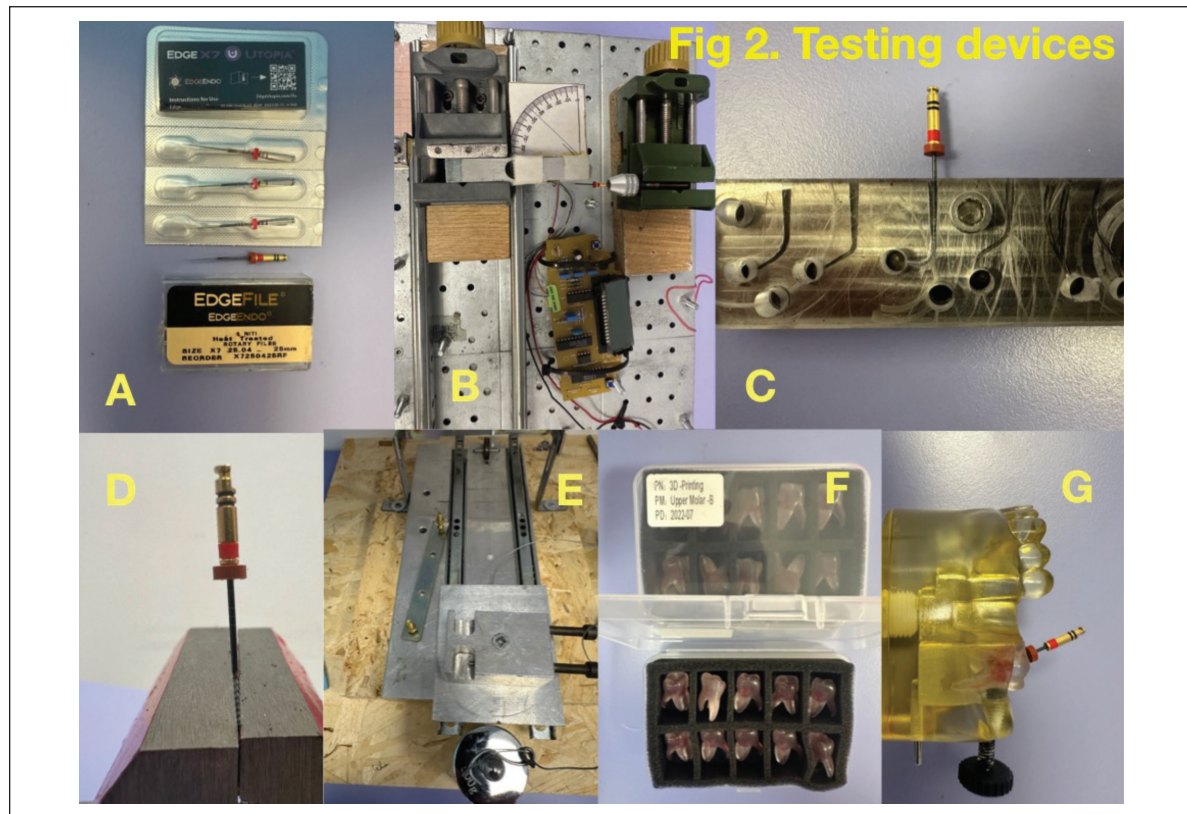


Figure 2.

mm from the tip of each instrument (max diameter = 0,49 mm) and instruments were rotated at 350 rpm and 2,5 N torque setting for 30 seconds. Each instrument was tested in linear cutting unidirectional lateral motion and the maximum penetration depth of the instruments was the criterion for cutting. Each plastic block was used to test one instrument from each of the two groups tested. The precise length of the plastic block cut in 1 min was measured in mm for all groups tested using a computerized program (Adobe Photoshop CS4) with a precision of 0.1 mm. The 1 mm notch was subtracted to the length obtained. Maximum penetration depth was calculated, mean and standard deviations of each group were calculated and data were statistically analyzed with a one-way ANOVA test with significance set at 95 % confidence interval.

Durability test

Each instrument was used to prepare five artificial 3d plastic (Fig 2f) molar tooth (Orodeka, Firenze, Italia) with the same motor and the same parameters (350 rpm

speed and 2,5 N torque). Overall, each instrument was supposed to prepare 20 canals (19) to working length without any breakage or deformation of flutes (fig 2g). An initial manual glide-path using a manual k-file n.15 was performed to ensure patency and a slight preliminar enlargement to facilitate the .04 25 instruments progression to the working length. All canals were prepared by the same expert clinicians using Mimeraci technique in steps: manual insertion, activation and progression in small steps (1-2 mm), removal of the file from canal, cleaning of flutes and irrigation with distilled water. Each step of the technique was repeated till working length was reached. The total instrumentation time, the incidence of instruments' separation or deformation of flutes (under microscope inspection at x10 magnification) were recorded. For the instrumentation time mean and standard deviations of each group were calculated and data were statistically analyzed with a one-way ANOVA test with significance set at 95 % confidence interval. Data concerning separated or deformed instruments were only recorded.

Table 1. Mean (standard deviation) results of different tests for the two instruments

Test	Parameters	x7 firewire	x7 utopia	P-values
Stiffness	maximum load	131,3 +/- 9,2	84, 2 +/- 6,5	<.001
Cyclic fatigue	time to fracture (s)	19,6 +/- 2,9	20,3 +/- 3,9	.412
Torsional resistance	Maximum torque (N. cm)	1, 34 +/- 0,28	1.01 +/- 0,19	<.001
Cutting ability	Penetration (cm)	13,4 +/- 2,6	9,8 +/- 3,9	<.001
Instrumentation time	Seconds (s)	12,9 +/- 7,7	16,1 +/- 9,2	<.001
Intracanal breakage	number of instruments	0	0	NA
Flute Deformation	number of instruments	4	0	NA

Results

Results are summarized in table 1. For the stiffness test X7 Utopia were found to be more rigid, with a statistically significant difference when compared to X7 Firewire. X7 Utopia were also found to be more resistant to torsion, showing significantly higher values for maximum torque at failure when compared to X7 Firewire, while no statistically significant difference was found between the two tested instruments when subjected to a cyclic fatigue test. For both torsional and cyclic fatigue tests no significant differences were noted in the two groups concerning fragment lengths, demonstrating a correct testing procedure. Under the conditions of the present test, cutting efficiency was significantly higher for X7 Utopia instruments, and also instrumentation time was significantly shorter when compared with X7 Firewire. During durability tests all instruments were able to reach working length without any intracanal breakage. On the contrary four X7 Firewire instruments which exhibited visible signs of flute deformation were discarded and were not able to prepare all the 20 canals, while no X7 Utopia instrument showed any sign of plastic deformation.

Discussion

The results of the present study showed that thermal treatment of nickel-titanium endodontic instruments involving intricate processes can significantly impact all their in vitro mechanical properties (flexibility, strength and cutting ability) and overall performance in root canal procedures (10, 20). Even if many factors contribute to the success of endodontic therapy, root canal instrumentation has a relevant role, because it create a proper shape to perform both final irrigation and obturation procedures correctly (21-25). Understanding the in vitro differences in thermal treatments is crucial for manufacturers to provide instruments with different properties and dental professionals to tailor these instruments to specific clinical requirements (1,5). In the present study two different heat treatments from the same manufacturer and applied to the same instrument design were tested and results showed significant differences between the two groups. As dimensions and designs were same these difference are only related to the difference in the heat treatments. Heat treatment typically involves processes like austenitization, quenching, and aging (9). Austenitization involves heating the NiTi alloy to a specific temperature to transform it from a martensitic to an austenitic phase. Subsequent quenching rapidly cools the alloy, fixing the desired phase and enhancing properties like shape memory. Aging, the final step, optimizes the balance between hardness and toughness. Variations in time, temperatures, heating and cooling processes may differentiate the heat treatments and ideally these advanced techniques should allow for precise control over specific attributes, offering a more tailored approach to performance (5). Unfortunately heat treatments are proprietary and not disclosed by manufacturers in details. Manufacturers only state the improvements in clinical or in vitro performance of the commercial instruments by mentioning the different applied HTs. They usually focus only on a few main properties, avoiding to mention all the differences (advantages and disadvantages) in performance related to these changes (10). As a consequence, clinicians are not aware of all proerties which could affect clinical performance in a positive or negative way (2).

Differences in thermal treatments of NiTi endodontic instruments stem from variations in methods and objectives (10). A relevant topic is in the pursuit of a balance between flexibility and resistance to cyclic fatigue. Heat treatment seeks to achieve this equilibrium through careful control of temperature and time during the thermal processes. The challenge is to prevent excessive hardness that could compromise flexibility while ensuring sufficient toughness to resist cyclic loading, by creating gradient structures within the material to optimize both flexibility and fatigue resistance.

In the present study all the tested instruments had a similar resistance to a cyclic fatigue test which was performed in a very challenging complex ,abrupt apical curvature.

Differences in the thermal treatment protocols contribute to the development of NiTi instruments with specific characteristics for various clinical applications (1,7). For instance, instruments designed for shaping procedures may undergo thermal treatments that prioritize flexibility to navigate curved root canals efficiently. In contrast, instruments intended for more simple and rapid techniques may prioritize durability and cutting efficiency. Results from the present study confirmed the significant impact of the different heat treatments. They showed that the X7 Utopia were less flexible, but more resistant to torsion and efficient in cutting when compared to Firewire X7. This property may be also more helpful in retreatment cases, making removal of gutta-percha easier and faster. Moreover less plastic deformation of flutes were observed after clinical use, leading to more durability.

The question whether such plastic deformations are a weak point or not is still open. Obviously permanently deformed rotary instruments should be discarded (which negatively affects durability), but such feature is also considered beneficial, since it is a clinical warning that could prevent sudden, unexpected intracanal failure. It is considered a warning because such plastic deformations usually occurs immediately prior to breakage.

In summary we may conclude that heat treatment methods provide a well-established positive approach, and new technologies offer more sophisticated and targeted modifications, like the differences shown between the two tested X7 instruments. Such avenues contribute to the ongoing evolution of NiTi rotary instruments, continually improving their performance and expanding their applicability in endodontic practice, ultimately benefiting dental practitioners and patients alike by providing tailored instruments for the case and overall by enhancing the efficiency and safety of endodontic shaping procedures.

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Contributions	Conceptualization	Methodology	Validation	Data Curation	Writing—Original draft	Writing—review	Supervision
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Author 2	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Author 3	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Author 4	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Author 5	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Author 6	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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