# Quantitative Techniques for Measuring Cleanroom Wipers with Respect to Airborne Molecular Contamination

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## ABSTRACT

Cleanroom wipers are generally of three classifications, namely, synthetic, natural fibers and blends of fibers. Ultra-fine split type fiber is mostly selected as the wiper material since cleanrooms need high filtering efficiency. The three wiper samples selected were made from knitted polyester filament fabric with hot-cut or sealed edges. The wiper samples A and B are considered for the application in Cleanroom Class 10 level environment and wiper sample C is for Cleanroom Class 100 level environment. ASTM E1560-95 and IEST-RP-CC004.3: 2004 are the two standards for testing cleanroom consumables. Following these standards, three specific wipers were compared in terms of residue concentration of liquid particles, ions, metals and non-volatile organic compounds using liquid particle counting (LPC), inductively coupled plasma mass spectrometry (ICP-MS), ion chromatography (IC), and nonvolatile residue (NVR) methods, respectively. All experiments were performed in a cleanroom of Class 100. Detailed test results were very informative to characterize the performance of wipers manufactured by specific processes and materials.

*Keywords:* Cleanroom; Wiper; Airborne molecular contamination; Particle; Ion; Nonvolatile residue.

## INTRODUCTION

In semiconductor and data storage devices manufacturing industries, it is of utmost importance to control even the minutest quantities of microcontaminants, such as particles and ions (Li *et al.*, 2007). It is now well understood that a microelectronic device can fail not only because of the presence of "killer particles," but also because of the ionic content in the particles. Ions such as Na, Li, NH4, K, Mg, Ca, F, Cl, NO4, PO4 and SO4 are generally inherently present in textile fabrics. These ions may be detrimental to a cleanroom environment, especially in the semiconductor industry, because the ions can get transferred to silicon wafer circuitry, causing corrosion and/or short circuiting on the wafer's circuitry (Bai *et al.*, 2002). It is known that deionized water can be used to reduce or eliminate these ions from the fabric for making it suitable for use in cleanroom applications. Deionized water attracts the ions in the fabric such that the ions are pulled off the fabric, into the

water, which can then be discarded or filtered for reuse. Typically, ion reduction or removal is achieved by using a cleanroom laundry to wash the fabric (Kim *et al.*, 2010; Yamada *et al.*, 2011), often in the form of wipers, to reduce ion content. However, this process is very expensive and time consuming and may detrimentally affect physical properties of the fabric since during the process of washing it often encounters overly aggressive agitation and rinsing and exposure to high temperature water and chemicals.

One critical source of ions in cleanrooms is consumables such as wipers used for controlling microcontamination. As wiping materials become cleaner, accurate measurement of minute quantities of ionic content in the wipers becomes an increasing challenge. As levels of ions in wipers are now well into the sub-ppm range, more sensitive and accurate measurement techniques are required to identify and quantify arrays of both cations and anions.

We evaluated and compared ions in three wiper samples. They represent several manufacturers of wipers primarily used in critical environments. All the selected wipers were made from knitted, polyester filament yarn, with hot-cut or border-sealed edges. Three wipers tested are identified in this article as products A, B and C. Test and measurement criteria of the tested wipers prescribed in IEST-CC00-

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RP4.3:2004 (2004) include particles ($\mu g/cm^2$), extractables (%), and ions (ppm). The three wiper samples were examined for the following characteristics:

- Releasable particles (in $\mu g/cm^2$).
- Releasable Li, Na, Mg, Al, K, Ca, Fe, F, Cl, Br, $NH_4$, $NO_4$, $PO_4$, and $SO_4$ (in micrograms per gram, which translates into parts per million).
- Nonvolatile residue (NVR) in de-ionized (DI) water (in %).

## MATERIALS AND METHODS

Four different test methods were used for evaluating three different wipers chosen to represent a spectrum of wipers marketed for use in critical environments. The three wiper samples selected were made from knitted polyester filament fabric with hot-cut or sealed edges, the specification of the wiper samples are listed in Table 1. For this research, three polyester cleanroom wipers were evaluated. All the experiments were done in a cleanroom (CLASS-100) with laminar flow benches. As described below, these wipers were exposed to a variety of test methods and environments during testing.

### Particle

The testing of cleanroom wipers has evolved over the past 20 years from simply shaking a material and visually approximating the amount of lint released to much more complex methods of separating particles from the wiper followed by quantification using sensitive analytical instrumentation. Controversies such as wet versus dry testing—removing particles via immersion in liquid as opposed to agitation in air, which predominated in the late 1980s, have today mostly been resolved in favor of wet testing (Mattina and Paley, 1990; IEST-RP-CC004.3, 2004). Published by the Institute of Environmental Sciences in June 1992, IES Recommended Practice 004 (Mattina and Paley, 1990) details two methods for removing particles from a wiper for subsequent enumeration using a laser-based liquid particle counter (LPC) or optical microscopy (Mattina and Paley, 1990). The first technique involves gently immersing the wiper in de-ionized (DI) water while the second method uses a more vigorous shaking of the wiper in a vessel containing DI water.

The test wiper was placed in a clean 6 cm × 32 cm × 46 cm (2.4 in. × 12.6 in. × 18.1 in.) polyethylene photographic tray filled with 500 ml of deionized water and a 25-mL aliquot of a 0.1 percent surfactant-based cleaning solution. The tray was then agitated using an biaxial shaker (Bhattacharjee and Paley, 1998; Webb, 2009) (Gilson, SS-15) at 150 rpm for 5 min. The biaxial shake test described in IEST-CC00-RP4.3 (2004) succeeds in imparting mechanical energy into the test environment as well as making the released particles available for counting in a liquid suspension. However, the test wiper must be folded or contorted to fit within the diameter of the shaker jar. Different wiping materials flex in different ways, and the initial wiper position in the jar can affect the final results. In contrast, if a laboratory orbital shaker is used for agitation, the wiper can be placed flat in a clean photographic tray similar to the one used for minimal stress tests. The tray is set onto the shaker and agitated at a set number of revolutions per minute for a fixed time. Because the wiper lies flat in the tray, its entire surface is always exposed to the effects of the mechanical agitation through the liquid. After agitation, the wiper was removed, the size was measured, and the liquid from the tray was poured into a clean 2-L beaker. To ensure that all the released particles and fibers were collected, the tray was rinsed with an additional 25 mL of deionized water, and the rinse water was added to the beaker. Contents of the beaker were filtered under vacuum using a 0.40-$\mu$m polycarbonate membrane filter. The filtration setup consisted of a stainless steel screen and a steel funnel, a teflon gasket, a spring clamp, and a vacuum pump capable of delivering minimum 50 torr of vacuum pressure. The filter was allowed to air dry and was then transferred to an aluminum specimen stub. The perimeter of the filter was affixed with several spots of conductive carbon paint. The last step of the sample preparation involved applying a thin layer of gold coating by using vacuum sputtering under an argon atmosphere. As part of the testing methodology, wipers were immersed in a 100% DI water solution which simulated the low surface-tension liquids often used in semiconductor manufacturing. Particles were measured by LPC (Liquid Particles Counter) with SEM (Scanning Electron Microscopy) (Bhattacharjee and Paley, 1997; Bhattacharjee and Paley, 1998; Webb, 2009).

With the experiment outlined above, the total number of particles that were generated includes the initial number of particles in the suspension, the number of particles released from the wiper during biaxial shaking, and the

**Table 1.** The specification of the wiper samples.

| Wiper Samples | Specification (Deviation 5%) | | |
|---|---|---|---|
| | A (Class 10) | B (Class 10) | C (Class 100) |
| Weight ($g/m^2$) | 125 | 150 | 145 |
| Thickness (mm) | 0.48 | 0.52 | 0.5 |
| Absorption ($g/m^2$) | > 350 | > 450 | > 450 |
| Gas Particle ($\geq 0.3$ $\mu$m) | < 100/cft | < 100/cft | < 100/cft |
| Liquid Particle ($\geq 0.5$ $\mu$m) | $< 10 \times E6/m^2$ | $< 10 \times E6/m^2$ | $< 10 \times E6/m^2$ |
| Non-volatile substance ($\mu g/cm^2$) | < 10 | < 10 | < 10 |
| Standard size, package | 150 pcs/Bag, | 150 pcs/bag, | 150 pcs/bag, |
| 9" × 9" Vacuum pack | 10 Bag/1Carton | 10 bag/1carton | 10 bag/1carton |

number of particles created from the break-up of larger aggregates. If the number of particles captured is negative, meaning that more particles were released into the water than removed, then the number of particles captured is defined as zero.

Therefore, the number of particles captured by the wiper is defined as (Peter, 2006):

Number of particles captured = initial particles in solution + particles released from wiper + particles created from particle break-up – particles after biaxial shake

Conversely, if the number of particles released is negative, then it is defined as zero since no particles were released, therefore

Number of particles released = particles after biaxial shake – particles in wet wiper – particles released from clean wiper.

### *Ion*

Metals and ions can cause significant harm to semiconductor devices and their manufacturing processes in many ways. Metals in the form of particles can become inadvertent dopants or form conductive bridges across circuit lines. In addition, as with any particle, metal particles can interfere with photolithography processes, making their detection and analysis doubly important. Anions are not only conductive, but can also cause corrosion in metalized layers. This is particularly detrimental because it is a reliability issue, and the device can fail long after final test. Both cations and anions can be released through particles or in moisture exposed to the wiper surface. Extraction of ions from the wiper in liquid and subsequent measurement with analytical instrumentation can quantify the burden of ions on the wiper surface.

Ions are the quantified species of matter that can be extracted from a wiper and are generally expressed as ppm or ppb. While the extraction method could use elevated temperature, most, if not all, wiper manufacturers use an ambient temperature soak for extraction (Peter, 2006). The advent of high-sensitivity analytical instrumentation (Webb, 2009) such as ion chromatography (IC), with its multi-ion separation and quantification capability, permits rapid and straight forward ion analyses at sub-ppm levels on a single extract (Webb, 2009). Capillary ion electrophoresis (CIE) can also be used for anion and cation determinations, albeit at lower sensitivity. Cations alone can be determined with graphite furnace atomic absorption spectrometry (GFAAS) or inductively coupled plasma mass spectrometry (ICP-MS) (Fang *et al.*, 2011). Of the techniques cited here, IC is the lowest in cost and, because of its versatility and sensitivity, it has become the most popular approach. This study used Ion Chromatography (IC) (ITW Texwipe, 2001; Siegerman, 2004; Ku *et al.*, 2010; Dionex, Application Note 113) and inductively coupled plasma mass spectrometry (ICP-MS) (Dionex, Application Note 94) as the two methods to measure the extracted ion content.

The samplers are typically self-contained and consist of acalibrated vacuum pump, Teflon impingers (SKC Model 225-0021, USA Headquarters International Sales SKC Inc.),

stainless steel tubing and a flow meter. The impingers are charged with a dilute acid solution used to capture metallic species and ultrapure water (UPW) to capture ionic species, with impingers typically arranged in three series to monitor recovery. This test method describes a deionized water extraction procedure for determination of Li, Na, Mg, Al, K, Ca and Fe, and supplies details on eluents used for each of these categories. Since transition metals are only sparingly soluble in water, a dilute acid mixture is used to extract them from wipers. After extraction, the transition metal cations can be separated in the IC system (DX-100, Dionex, Sunnyvale, CA, USA)using pyridine-2,6-dicarboxylic acid (PDCA)as a complex eluent, and subsequently determined as the post column eluent 4-(2-pyridylazo) resorcinol (PAR) complex esusing a conductivity detector.

ICP-MS is a combination of an inductively coupled plasma (ICP) with a mass spectrometer (MS).The instrumentation included a flow injection X7 ICP-MS instrument (Thermo Electron, Waltham, MA, USA) equipped with a sample introduction system, an ICP torch comprised of three concentric quartz tubes, a quadrupole mass analyzer, a simultaneous analogue/pulse counting electron multiplier detector and an ASX-510 HS auto sampler (CETAC, Omaha, NE, USA). The sample introduction system consisted of a close-coupled, variable speed, three-channel peristaltic pump, a glass concentric nebulizer and a glass single-pass conical spray chamber with a fixed impact bead.

The sample is introduced into the ICP by a sample introduction system consisting of a peristaltic pump and an ebulizer, which generates a fine aerosol in a spray chamber. The spray chamber separates the small droplets from the large droplets. Large droplets fall out by gravity and exit through the drain tube at the end of the spray chamber, while the small droplets pass between the outer wall and the central tube and are eventually transported into the sample injector of the plasma torch using a flow of argon gas. The aerosol is then transported to the ICP, which is a plasma ion source. This plasma is formed by the application of a high voltage spark to a tangential flow of argon gas, which causes electrons to be stripped from their argon atoms. These electrons are caught up and accelerated into a magnetic field, formed by a radio frequency (RF) energy which is applied on a RF coil surrounding the plasma torch. This process causes a chain reaction of collision-induced ionization leading to an ICP discharge. The ICP reaches temperatures of 6,000–8,000 K. As the aerosol transits the plasma, the droplets undergo numerous processes which include desolvation, dissociation, atomization, and ionization (Balazs, 1995). Ions produced by the argon ICP are principally atomic and singly charged, making it an ideal source for atomic MS. Since the ICP works at atmospheric pressure and the MS requires a vacuum, an interface typically consisting of a coaxial assembly of two cones (sampler and skimmer cone) and a series of pressured differentials to allow efficient sampling of the atmospheric pressure plasma gases while minimally perturbing the composition of the sample gases. After passing through the sampler and skimmer cones, several electrostatic lenses or ion optics focus the ions into the MS,

where the ions are separated based on their mass-to-charge (m/z) ratios. After passing the mass separator, the ions strike the active surface of the detector, typically an electron multiplier. Each ion which hits the channel electron multiplier generates a cascade of electrons leading to a discrete pulse. The pulses are counted and the output signal is given in counts per second.

### Nonvolatile Residues

Nonvolatile residue can be extracted from a wiper when it is used with a solvent. This residue remains on a wiped surface, leaving it coated with an organic film. Many wipers contain high levels of chemical additives to achieve specific performance characteristics. These additives, which are soluble in common solvents, are often deposited on clean surfaces that have been wiped with the wiper. Fortunately, numerous techniques exist for detecting the presence of potential extractables. There are several tests available for measuring and analyzing nonvolatile residue released from wipers in a given solvent. To test for NVR (Jarvis *et al.*, 1992; ASTM E1560-95, 2001) in DI water, three wiper samples were boiled in water for 5 minutes. After boiling, the liquid was filtered, the filtrate was evaporated to dryness, and the residue weighed. Weighing must be with a very sensitive analytical balance, as weight of residue is often very small, measurable in tenths of a milligram. The residue can then be collected and weighed. The results were normalized by dividing by the area of the wipers (in %).

### RESULTSANDDISCUSSION

### Liquid Particles Counts

Each of the sample preparation techniques was evaluated using multiple samples of each wiper to ensure consistency of data. The particle count results were examined in terms of differences between wipers for the test method as well as for variation in particle counts from the test method. The results are classified by size into two categories: small particles between 0.5 μm and 5μm; and large particles greater than 5 μm but smaller than 100 μm. The use of scanning electron and optical microscopes as viewing and enumeration tools provides a direct and precise measurement of the quantity and type of contamination. Large and small particles were counted using a SEM at 200X and 3000X magnifications, respectively. At 200X, computer-aided image analysis and counting were used. At 3000X, counting was performed either manually or automatically, depending on the number of particles per field of view (Paley, 1996). The data were then subjected to statistical analysis with an objective to achieve ± 10-percentaccuracy at a confidence level of 95 percent.

As shown in Table 2, the test results showed that wiper sample C of Cleanroom Class 100 level released over 3–7 times more small particles than wiper samples A and B of Cleanroom Class 10 level. Potentially, these released particles could cause recontamination or cross-contamination issues. The test results for large particles showed that

**Table 2.** Release of A.C. Fine Test Dusts particles for three wiper samples.

| Particle Diameter(μm) | Liquid Particle Counts (μg/cm$^2$) | | |
|---|---|---|---|
| | A | B | C |
| 0.3–0.5 | 44937.2 | 40632.2 | 303904.2 |
| 0.5–1.0 | 4557.6 | 4546.2 | 44493.1 |
| 1.0–5.0 | 81.2 | 190.6 | 849.9 |
| > 5.0 | 2.5 | 8 | 22.7 |

wiper sample C of Cleanroom Class 100 levelreleased3-9 times more particles than wiper samples A and B of Cleanroom Class 10 level. Based on an analysis of the ratio data, it can be concluded that the most effective mechanism for releasing particles from wipers involves both mechanical agitation and the immersion of a wiper in a low-surface-tension environment; this also most closely approximates conditions during use.

### Heavy Metal Concentration Analysis by ICP-MS

The data resulting from the analysis of a sample consists of the number of ions counted at fixed m/z. Normally the total ions counting are converted to ions counted per second (cps) to give counts. The analyte signal depends on how many ions are counted by the detector and this is influenced by a number of factors. The total number of atoms available in the sample will depend on the abundance of the isotope measured. The percentage of atoms that are ionized depends on the ionization energy of the element. However, even when these factors are corrected, elements of differing mass do not produce the same signal for the same concentration. Coupled with inherent mass bias caused by ion extraction and transmission, the ICP has a high degree of spatial resolution and ions are not homogeneously spaced throughout (Niu and Houk, 1996).

Total heavy metal concentrations extracted from the three wiper samples at normal temperature (25°C) and high temperature (80°C) are summarized in Tables 3and 4, respectively. Concentrations of Li, Na, Mg, Al, K, Ca and Fe were examined. Total heavy metal concentrations at high temperature were significantly higher than those at normal temperature. At normal temperature, some total

**Table 3.** Concentrations (μg/g) of heavy metals in three wiper samples collected at normal temperature.

| | Li | Na | Mg | Al | K | Ca | Fe |
|---|---|---|---|---|---|---|---|
| Wiper A | BDL | 0.264 | 1.97 | BDL | 0.27 | 7.76 | 0.019 |
| Wiper B | BDL | 0.38 | 0.051 | BDL | 0.26 | 7.43 | 0.008 |
| Wiper C | BDL | 6.26 | 4.26 | BDL | 1.88 | 20.2 | 0.026 |

**Table 4.** Concentrations (μg/g) of heavy metals in three wiper samples collected at high temperature.

| | Li | Na | Mg | Al | K | Ca | Fe |
|---|---|---|---|---|---|---|---|
| Wiper A | BDL | 0.38 | 2.02 | 0.025 | 0.303 | 11.8 | 0.029 |
| Wiper B | 0.015 | 0.32 | 0.043 | 0.005 | BDL | 8.47 | 0.008 |
| Wiper C | BDL | 6.44 | 4.69 | 0.059 | 2.61 | 23.32 | 0.033 |

Note. BDL: Below Determination Limit.

micro-metal concentrations are lower than the detection limit of the instrument, but they can be detected at high temperature. In particular, wiper sample C of Cleanroom Class 100 level was highly enriched compared with wiper samples A and B of Cleanroom Class 10 level, indicating that dominant quantities of these elements are derived from water rinse.

### Anions and Cations Concentration Analysis by IC

In this analytical method (Kathryn and Kym, 2009), the sample analyte is swept through an analytical column, filled with an appropriate stationary phase, by an eluent. Separation of the ionic constituents is achieved through different retention times, depending on their binding energy and size, with the stationary phase for each ion. Pre-concentration of the sample is necessary to achieve sensitivity to the low ppt levels. After pre-concentration, the solution (analyte and eluent) passes through a separation unit to reduce eluent conductivity to a very low level. The analyte finally continues through to a conductivity detector to generate a signal (Tan, 1998; Victor, 2002).

Tables 5–8 show results of extracted DI water extractable anions and cations of the three wipe samples. For the purpose of this discussion, only selected anions and cations from the suite of ions analyzed ($F^-$, $Cl^-$, $NO_2^-$, $Br^-$, $SO_4^{2-}$, $PO_4^{3-}$, $Li^+$, $Na^+$, $NH_4^+$, $K^+$, $Mg^{2+}$, $Ca^{2+}$) are shown in the table. As shown in Tables 5–8, test results showed that concentrations of anions extracted from wiper sample B of Cleanroom Class 10 level were lower than the detectable limit. Concentrations of anions and cations extracted from wiper sample C of Cleanroom Class 100 level were very high, 10 times more than wiper samples A and B of Cleanroom Class 10 level. In high temperature conditions, anions and cations are extracted easily from the same sample.

### Nonvolatile Residues

The quantity of residue extracted is dependent upon the mass of wiper tested and the quantity of solvent used. Select a mass of material that is likely to provide at least 0.0020 g of extracted residue. If the analysis yields less than 0.0020 g of residue, repeat the analysis with a higher mass of material. Obviously, there is a limit to the weight of material that can reasonably be processed utilizing this procedure. In general, no more than 50 grams of wiper need be extracted in an effort to meet the minimum mass of extracted residue.(TW Texwipe, 2008) If, due to the nature of the wiper, sufficient material cannot be extracted to meet the minimum requirement, test the largest sample mass that can reasonably be processed. As seen in Table 9, wiper sample B in Cleanroom Class 10 had very low NVR value (0.108%). Wiper sample C of Cleanroom Class 100 level had very high NVR value (1.196%), 10 times higher than wiper samples A and B of Cleanroom Class 10 level.

## CONCLUSIONS

To provide useful risk-assessment data, testing of consumable materials that are brought into cleanroom environments needs to closely simulate the conditions of

**Table 5.** Extracted anions concentration at normal temperature.

|  | $F^-$ | $Cl^-$ | $Br^-$ | $NO_2^-$ | $PO_4^{3-}$ | $SO_4^{2-}$ |
|---|---|---|---|---|---|---|
| Wiper A | BDL | 0.445 | BDL | 0.289 | BDL | 0.823 |
| Wiper B | BDL | BDL | BDL | BDL | 0.362 | 0.34 |
| Wiper C | BDL | 5.02 | BDL | 0.677 | 0.119 | 10.44 |

Note. BDL: Below Determination Limit.

**Table 6.** Extracted anions concentration at high temperature.

|  | $F^-$ | $Cl^-$ | $Br^-$ | $NO_2^-$ | $PO_4^{3-}$ | $SO_4^{2-}$ |
|---|---|---|---|---|---|---|
| Wiper A | BDL | 1.023 | BDL | 0.601 | 0.111 | 1.046 |
| Wiper B | 0.17 | 0.255 | BDL | 0.255 | 1.255 | 0.383 |
| Wiper C | BDL | 6.532 | BDL | 1.155 | 0.438 | 11.073 |

Note. BDL: Below Determination Limit.

**Table 7.** Extracted cations concentration at normal temperature.

|  | $Li^+$ | $Na^+$ | $NH_4^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ |
|---|---|---|---|---|---|---|
| Wiper A | 0 | 0.311 | BDL | BDL | 2.47 | 14.66 |
| Wiper B | 0 | 0.511 | 0.319 | 0.362 | 0.043 | 9.92 |
| Wiper C | 0 | 0.6213 | 0.996 | 2.67 | 5.74 | 27.32 |

Note. BDL: Below Determination Limit.

**Table 8.** Extracted cations concentration at high temperature.

|  | $Li^+$ | $Na^+$ | $NH_4^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ |
|---|---|---|---|---|---|---|
| Wiper A | 0.001 | 0.36 | BDL | BDL | 2.58 | 18.17 |
| Wiper B | 0.021 | 0.68 | 1.3 | BDL | 0.043 | 11.21 |
| Wiper C | 0 | 8.6 | 6.21 | 1.67 | 6.37 | 30.67 |

Note. BDL: Below Determination Limit.

**Table 9.** Results of the test for NVR.

| Sample Number | A | B | C |
|---|---|---|---|
| NVR (%) | 0.219 | 0.108 | 1.196 |

use. The goal of the research described here was to explore the design of such a test for cleanroom wipers. Improvements in wiper manufacturing technology have been demonstrated to produce lower levels of releasable particles, micro-metals, anions and cations, and nonvolatile residues. Liquid particle counts, ICP-MS, IC and NVR solvent methods were used to compare samples of three different knitted polyester cleanroom wipers. Using these measurement tools, improvements have been made in reduction of particles, micro-metals, anions and cations, and nonvolatile residues released from wipers through unique wiper constructions, edge sealing, and laundering processes. The ideal wiper, however, should not only start out clean but be able to capture and hold contaminants until they can be taken out of the clean environment and discarded. Tested sample wipers of A and B were found to perform 10 times better than the sample wiper C.

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