Hydrogen peroxide ($\text{H}_2\text{O}_2$) is a vital metabolite in the physiological system which regulates cellular signals, renal functioning, stem cell proliferation, protein synthesis and also kills the invaded pathogens.\textsuperscript{1,2} Owing to the strong oxidizing/reducing properties, as well as ease of availability, storability and facile handling, $\text{H}_2\text{O}_2$ has become an indispensable candidate in various domains like clinical (antiseptic/disinfectant), industrial (bleaching), food (processing, packaging and cleaning) and environmental (waste management and water treatment) fields.\textsuperscript{3,4} Besides, $\text{H}_2\text{O}_2$ is the primary reactive oxygen species (ROS) that is produced naturally in mitochondrial organelles by most of the oxidases for effective cell functioning.\textsuperscript{5,6} The difference between the produced ROS and regulated ones in cells is known as oxidative stress.\textsuperscript{7} Therefore, $\text{H}_2\text{O}_2$ has become a desirable biomarker for several oxidative stress-induced diseases like diabetes, cardiovascular, cancer and neurodegenerative diseases like Alzheimer’s and Parkinson’s.\textsuperscript{10-12} Hence, it is a compelling need to develop a highly efficient, selective and sensitive method for its real-time detection.\textsuperscript{13} Among various existing techniques such as spectrophotometry, colourimetry, fluorometry, titrimetry, electrochemical, chemiluminescence and chromatography, the electrochemical methods are extensively adopted owing to their sensitivity, portability, simplicity, repeatability, rapid response time and cost-effectiveness.\textsuperscript{14-17} The electrochemical sensing of $\text{H}_2\text{O}_2$ can be done either by enzymatically or non-enzymatically fabricated electrodes.\textsuperscript{18} Even though both the approaches have proven their effectiveness, enzyme-based electrodes have some challenges like less operational and storage stability, high cost of the enzyme, sensitive to harsh conditions, complicated fabrication process, etc., and hence, restricts their practical applicability.\textsuperscript{19-21} On the other hand, non-enzymatic sensors exhibit high stability, cost-effectiveness, longer storage period and can be reused multiple times without the loss in their activity.\textsuperscript{22} Therefore, significant effort has been dedicated to the development of enzyme-free sensors with improved electrocatalytic activities through identifying and designing promising electrode materials and/or their fabrication methodologies.

The choice of electrode material plays a significant role in the fabrication of a non-enzymatic sensor since these materials dramatically influences the sensitivity, selectivity, catalytic activity and stability of the sensor.\textsuperscript{23} Therefore, a variety of materials are continuously being explored, which includes carbon nanomaterials, 2D materials, metal nanoparticles, metal oxides, complexes, polymers, ionic liquids (ILs) and so on.\textsuperscript{24-26} Among them, IL based electrochemical sensors and biosensors are increasingly reported owing to their unique characteristics including excellent ionic conductivity, synthetic versatility, good electrochemical stability, less toxicity and tunable physicochemical properties.\textsuperscript{27-32} Further, even if IL structure comprises some toxic moieties, they can be suitably derivatized to non-toxic forms using their synthetic versatility and can be disposed safely. Incorporation of a redox mediator into the IL matrix would facilitate the electrocatalytic process through their electron transferability, which in turn would amplify the analytical performance of the sensors.\textsuperscript{33,34} Accordingly, we envisioned to design and develop a hybrid system comprising of redox mediator and IL, which would beneficially exhibit the characteristics of both of them.\textsuperscript{35}

Based on the above perspective, we have prudently designed and synthesized an IL tagged anthraquinone and fabricated a disposable electrochemical sensor for the selective determination of $\text{H}_2\text{O}_2$. Anthraquinone and its derivatives are a prominent class of redox species, owing to their rich $\pi$-electrons and reversible redox couple of quinone/hydroquinone.\textsuperscript{36,37} The stable redox behaviour as well as the redox potential of AQ motivated us to utilize it as suitable redox mediator in electrode fabrication. More importantly, the potential tunability of ILs owns a particular interest in sensor design for tuning the redox properties (usually redox potential) of redox mediators.\textsuperscript{38} and thus, ILs were sensibly introduced in the AQ structure to further reduce the redox potentials of AQ and to enhance the conductivity of the electrode design. Thus synthesized AQ-PF$_6$-IL was deposited over a screen printed electrode (SPE) to
obtain AQ-PF$_6$-IL/SPE modified electrode, which on electrochemical investigation revealed excellent redox peaks of AQ with reduced formal potential compared to AQ/SPE. Further, the fabricated AQ-PF$_6$-IL/SPE was successfully applied as a disposable non-enzymatic sensor towards the sensitive and selective detection of H$_2$O$_2$.

**Experimental**

**Synthesis of 1-benzyl-3-(9,10-dioxo-9,10-dihydroanthracen-2yl)methyl)-1H-imidazol-3-ium chloride (AQ-Cl-IL).**—The procedure for the synthesis of benzyl-1H-imidazole is detailed in the supporting information (SI) and the stepwise synthesis of AQ-Cl-IL is presented in Fig. 1. The redox mediator, anthraquinone (0.256 g, 1 mmol) in toluene. After completion of the addition, the resulting mixture has been refluxed for 16 h at 75 °C. The solid product was separated by filtration and was washed twice with toluene and diethyl ether, then dried to acquire AQ-Cl-IL 1H NMR with toluene and diethyl ether, then dried to acquire AQ-Cl-IL (Yield 92%).

**Synthesis of 1-benzyl-3-(9,10-dioxo-9,10-dihydroanthracen-2yl)methyl)-1H-imidazol-3-ium chloride (AQ-Cl-IL).**—The procedure for the synthesis of benzyl-1H-imidazole is detailed in the supporting information (SI) and the stepwise synthesis of AQ-Cl-IL is presented in Fig. 1. The redox mediator, anthraquinone (0.256 g, 1 mmol) in toluene. After completion of the addition, the resulting mixture has been refluxed for 16 h at 75 °C. The solid product was separated by filtration and was washed twice with toluene and diethyl ether, then dried to acquire AQ-Cl-IL 1H NMR with toluene and diethyl ether, then dried to acquire AQ-Cl-IL (Yield 92%).

**Results and Discussion**

**Characterization of synthesized AQ-PF$_6$-IL.**—The chemical purity of the synthesized AQ-CI-IL and AQ-PF$_6$-IL were ascertained with $^1$H, $^{13}$C, $^{31}$P, $^{19}$F NMR and ESI mass analysis (Figs. S1–S8). Further, the UV–vis spectral measurements of AQ (black), AQ-CI-IL (red) and AQ-PF$_6$-IL (blue) were recorded in dichloromethane (1 $\times$ 10$^{-5}$ M) and are displayed in Fig. 2A. The sharp peak and a broad band at 248 and 323 nm correspond to the π–π$^*$ transitions of benzenoid and quinonoid units present in the AQ molecule, respectively.39 Interestingly, there were no apparent changes in the absorbance of AQ after tagging with IL (AQ-CI-IL, AQ-PF$_6$-IL), which reveals that incorporation of IL does not alter the structure of AQ and shows great compatibility with AQ. Furthermore, FTIR was performed to investigate the functional changes that have occurred during IL formation and the obtained data are shown in Fig. 2B. In AQ spectrum, the band at 1675 cm$^{-1}$ corresponds to the stretching vibration of dicarbonyl quinone unit and the band at 1594 cm$^{-1}$ represents the C–C stretching frequency of aromatic rings. Both the above-mentioned peaks were sustained even after IL formation, which also denotes that AQ retains its characteristics in the IL form. In addition, the sharp peak that appeared in AQ-PF$_6$-IL at 836 cm$^{-1}$ corresponds to the stretching vibrations of P–F (PF$_6$ anion); however, the absence of P–F stretching in AQ-CI-IL and AQ confirms the successful anion exchange from chloride to hexafluorophosphate anion in AQ-PF$_6$-IL.40 These results clearly demonstrate that the structure of AQ was not altered even after IL formation, and hence, the mediating property of AQ is conserved in ionic liquid form of AQ-PF$_6$-IL.

**Fabrication of AQ-PF$_6$-IL/SPE.**—Before electrode modification, the SPE was thoroughly washed with water and then activated electrochemically by anodizing at +2.0 V for 500 s in 0.1 M H$_2$SO$_4$ under constant stirring. After anodization, the SPE was rinsed with water and dried under nitrogen current. Thereafter, 10 μL of AQ-PF$_6$-IL (1 mg/100 μL of dichloromethane) was drop coated on the preanodized SPE and then dried at RT to form AQ-PF$_6$-IL/SPE. For comparison, AQ/SPE was fabricated similarly by drop-casting AQ (1 mg/100 μL of dichloromethane) on SPE. The synthesis of AQ-Cl-IL was accomplished by the slow addition of toluene solution of synthesized 1-benzyl-1H-imidazole (0.158 g, 1 mmol) to the stirring reaction mixture of 2-(chloromethyl)anthraquinone (0.256 g, 1 mmol) in toluene. After completion of the addition, the resulting mixture has been refluxed for 16 h at 75 °C. The solid product was separated by filtration and was washed twice with toluene and diethyl ether, then dried to acquire AQ-Cl-IL 1H NMR with toluene and diethyl ether, then dried to acquire AQ-Cl-IL (Yield 92%).

**Electrochemical impedance spectroscopy on AQ-PF$_6$-IL/SPE.**—To investigate the electrochemical properties of the fabricated sensor, electrochemical impedance spectroscopy (EIS) was executed on different modified electrodes to understand the surface modification through impedance change that has occurred during the electrode construction. Figure 3A represents the Nyquist plot obtained for bare SPE (black), AQ/SPE (red) and AQ-PF$_6$-IL/SPE (blue) in 0.1 M KCl with 2 mM Fe[(CN)$_6$]$_3$−/4− solution. The charge transfer resistance ($R_\text{ct}$) of the bare SPE was calculated to be 359 Ω, which denotes the inadequate electron transfer process at bare SPE. However, immobilization of AQ on SPE reduces the $R_\text{ct}$ value to 162 Ω, which implies that modification with AQ (AQ/SPE) improves the

Figure 1. Synthesis of 1-benzyl-3-(9,10-dioxo-9,10-dihydroanthracen-2yl)methyl)-1H-imidazol-3-ium hexafluorophosphate (AQ-PF$_6$-IL).
electrochemical properties of SPE. Interestingly, after incorporation of IL into AQ (AQ-PF6-IL/SPE) the $R_{ct}$ value significantly reduced to 98 $\Omega$, representing the superior conductivity of the developed sensor. The predominant decrease in the $R_{ct}$ value of AQ-PF6-IL/SPE could be accredited to the conducting nature of IL, which enhances the electron transfer process thereby, improving its electrochemical properties.

**Electrochemical behaviour of AQ-PF6-IL/SPE.**—The redox characteristics of the developed AQ-PF6-IL/SPE was realized through cyclic voltammetry. Figure 3B displays the voltammetric response of bare SPE (black), AQ/SPE (red) and AQ-PF6-IL/SPE (blue) in 0.1 M phosphate buffer solution (PBS, N2 saturated) in the potential window from $-0.1$ to $-0.7$ V (sweep rate of 50 mV s$^{-1}$).

As anticipated, the unmodified SPE (bare) has not shown any redox behaviour in the scanned potential range, which ensures the absence of electroactive species on the bare SPE. On the other hand, native anthraquinone modified electrode (AQ/SPE) displayed a redox peak corresponding to anthraquinone/anthrahydroquinone with an anodic potential ($E_{pa}$) at $-0.43$ V, cathodic potential ($E_{pc}$) at $-0.67$ V and a formal potential ($E^0$) at $-0.55$ V. Interestingly, the fabricated AQ-PF6-IL/SPE showed a strong redox response with higher peak currents and reduced peak potentials of $E_{pa}$ and $E_{pc}$ at $-0.44$ and $-0.52$ V ($E^0 = -0.48$ V) compared to bare SPE and AQ/SPE. The superior behaviour of AQ-PF6-IL/SPE is accredited to the integration of IL into the sensor setup, which enhanced their electrochemical properties by improving their peak currents as well as reducing their peak separation and formal potential.

**Effect of sweep rate and pH on AQ-PF6-IL/SPE.**—The influence of sweep rates on the electrochemical behavior of the developed sensor was evaluated by performing cyclic voltammetry at various sweep rates from 5–200 mV s$^{-1}$ in N2 saturated 0.1 M PBS and the obtained voltammograms are shown in Fig. S10A. On incremental sweep rates, both the anodic and cathodic peak currents were gradually increased with a minimal shift in the peak potentials at a higher sweep rate. The plot of peak current vs square root of sweep rate produced a linear response, representing a diffusion-controlled process at the AQ-PF6-IL/SPE (Fig. S10B). Further, the impact of pH of the electrolyte on the fabricated sensor behavior was evaluated by recording the CV of AQ-PF6-IL/SPE in 0.1 M PBS at different pHs ranging from pH 5 to pH 10. At neutral pH, the redox peak current was maximum, and hence pH 7 was chosen as the optimum working pH for further electrocatalytic studies (Fig. S11). Further, the influence of pH on the peak potential of the sensor was investigated. The formal potential of the AQ-PF6-IL/SPE was found to vary with pH and a linear response was obtained with a slope of 55 mV pH$^{-1}$, corresponding to the theoretical value (59 mV pH$^{-1}$) of a two electron, two proton redox process.
Electrocatalytic response of AQ-PF₆-IL/SPE towards H₂O₂ reduction.—The impressive electrochemical behaviour of AQ-PF₆-IL/SPE inspired us to investigate the electrocatalytic ability of the developed sensor. Accordingly, the cyclic voltammetry was performed with AQ-PF₆-IL/SPE with increasing concentrations of H₂O₂ in 0.1 M PBS (pH 7.0) at 50 mV s⁻¹ (optimized conditions). Interestingly, for every addition of H₂O₂ (0.1–0.5 mM), the cathodic peak current linearly increased along with the concurrent decline in anodic peak current, which signifies the electrocatalytic H₂O₂ reduction at the newly fabricated sensor (Fig. 4B). Upon applying the reduction potential, anthraquinone gets reduced to anthrahydroquinone by electrochemical reduction (Fig. 4E). Thereafter, anthrahydroquinone reduces the spiked H₂O₂ into water and gets re-oxidized to anthraquinone, which can be confirmed by an increase in the reduction current as shown in Fig. 4B. This catalytic process continues as long as H₂O₂ available at the electrode surface.⁴¹,⁴² Further, to validate the efficacy of constructed sensor, the electrocatalytic performance was compared with other modified electrodes under optimized conditions. Figure 4A and its inset shows the CV response of AQ/SPE and bare SPE with 0.5 mM of H₂O₂ and it could be observed that there was a moderate increment in the reduction current of AQ/SPE at a higher reduction potential compared to AQ-PF₆-IL/SPE. Meanwhile, bare SPE showed a feeble current increase towards H₂O₂ reduction, which is evident only at larger reduction potential, revealing the poor catalytic behaviour of bare SPE and therefore, it is not suitable for H₂O₂ determination. Moreover, the current response of AQ-PF₆-IL/SPE was ~4 and ~9 folds higher than that of AQ/SPE and bare SPE.
respectively. These outcomes indicated that the electrocatalytic behaviour of AQ was unanimously improved as a result of IL formation (AQ-PF$_6$-IL) and thus, AQ-PF$_6$-IL/SPE showed remarkable current increase for the successive additions of H$_2$O$_2$, demonstrating the potential ability of the developed AQ-PF$_6$-IL sensor towards H$_2$O$_2$ reduction.

Amperometric determination of H$_2$O$_2$ at AQ-PF$_6$-IL/SPE.

—The superior voltammetric response of the developed sensor instigated us further to investigate their performance under dynamic conditions. Therefore, amperometry was performed with AQ-PF$_6$-IL/SPE (blue), AQ/SPE (red) and bare SPE (black) under optimized parameters with successive additions of H$_2$O$_2$ at an operating potential of −0.55 V into the constantly stirring (350 rpm) N$_2$ saturated 0.1 M PBS. For every addition of H$_2$O$_2$, the catalytic current was rapidly increased for AQ-PF$_6$-IL/SPE (blue) and stabilized within ∼3 s, representing the effective reduction of H$_2$O$_2$ at the fabricated sensor. Conversely, AQ/SPE and bare SPE showed lesser current response for the same concentrations of H$_2$O$_2$. Figure 4D portrays the plot of catalytic currents against the concentration of H$_2$O$_2$, in which AQ-PF$_6$-IL/SPE showed wide linear response over the concentration range from 10 to 1128 μM with sensitivity and limit of detection (LOD) as 0.281 μA μM$^{-1}$ cm$^{-2}$ and 2.87 μM (S/N = 3), respectively. The sensitivity has been calculated using the slope (m) obtained from the calibration plot divided by the area of the working electrode (0.196 cm$^2$). LOD was determined using the formula $3\sigma/m$, where $\sigma$ is the standard deviation obtained for 10 blank measurements. The obtained performance of the developed sensor was compared with the performances of recently reported H$_2$O$_2$ electrochemical sensors, which revealed that the fabricated sensor exhibited relatively better performance (Table I). The outstanding performance of AQ-PF$_6$-IL/SPE could be accredited to the integration of IL into the AQ structure (AQ-PF$_6$-IL), which enhanced the electrochemical properties by improving the electron transfer process, thereby improving the catalytic current and reducing the detection potential for the electrochemical reduction of H$_2$O$_2$.

Effect of interfering species on AQ-PF$_6$-IL/SPE.

—Anti-interference ability is the essential phenomenon for any enzyme-free sensor to evaluate its performance and practical applicability. Therefore, the influence of some potentially interfering chemical species that might be possibly present in real samples were prudently added during the H$_2$O$_2$ detection at AQ-PF$_6$-IL/SPE under optimized conditions. Figure 5A depicts the amperometry of AQ-PF$_6$-IL/SPE for the determination of 0.15 mM H$_2$O$_2$ along with the added during the H$_2$O$_2$ detection at AQ-PF$_6$-IL/SPE under optimized parameters with successive additions of H$_2$O$_2$ and it is found that the sensor was able to retain 96.27% and 93.65%, respectively, of the original current signal even upto 30 days. Further, to testify the reproducibility of the constructed sensor, the current responses of five identically fabricated AQ-PF$_6$-IL/SPE sensors were subjected to voltammetric measurements under optimized conditions with and without H$_2$O$_2$ (0.3 mM). Figure 5D shows the obtained current response of five AQ-PF$_6$-IL/SPEs, and their relative standard deviation (%RSD) were calculated to be 1.78% and 2.72% in absence and presence, respectively, revealing the good reproducibility of AQ-PF$_6$-IL/SPE towards H$_2$O$_2$ determination. The superior stability and reproducibility of AQ-PF$_6$-IL/SPE could be due to the formation of IL with AQ structure, which enhanced their electrochemical properties, thereby offering excellent cyclic and storage stability along with good electrode-to-electrode reproducibility.

Real sample analysis.

—The constructed AQ-PF$_6$-IL/SPE sensor was utilized for the detection of H$_2$O$_2$ spiked in real samples under optimized parameters by following a standard addition method to evaluate its feasibility in practical analysis. Accordingly, amperometry was performed with known concentrations of H$_2$O$_2$ spiked in diluted apple juice, and milk samples and the acquired responses are depicted in Fig. S12. The calculated recoveries were in the range between 96.54 and 102.12% and the %RSDs were in the acceptable range (Table II). These results substantiate that the fabricated ionic liquid based anthraquinone sensor has potential applicability in real-time monitoring of H$_2$O$_2$.

Conclusions

This investigation reports the fabrication of a disposable enzyme-free H$_2$O$_2$ electrochemical sensor using an IL tagged redox mediator modified electrode. Herein, we have designed and synthesized an IL tagged anthraquinone (AQ-PF$_6$-IL) for the first time and effectively utilized it in the fabrication of a modified electrode (AQ-PF$_6$-IL/SPE) to explore their electrochemical and electrocatalytic properties. The fabricated electrode exhibited a pair of distinct redox peaks attributable to anthraquinone/anthrahydroquinone redox pair. Moreover, IL formation improved the electrochemical properties of AQ thereby, the developed AQ-PF$_6$-IL/SPE sensor has remarkable selectivity for H$_2$O$_2$ determination.

Stability and reproducibility studies on AQ-PF$_6$-IL/SPE.

—To validate the potential stability of AQ-PF$_6$-IL/SPE, its electrochemical response was recorded over 50 continuous scans under optimized conditions and the obtained CVs are shown in Fig. 5C. There were no appreciable changes in the peak currents and potentials, indicating superior cyclic stability of the developed sensor. Further, the long-term stability of AQ-PF$_6$-IL/SPE was probed by quantifying its current response periodically for 30 days by storing it at RT. Inset to Fig. 5C depicts the obtained current response of AQ-PF$_6$-IL/SPE in the absence and presence of 0.3 mM H$_2$O$_2$ and it is found that the sensor was able to retain 96.27% and 93.65%, respectively, of the original current signal even upto 30 days. Therefore, the obtained performance of the developed sensor was compared with the performances of recently reported H$_2$O$_2$ electrochemical sensors, which revealed that the fabricated sensor exhibited relatively better performance. (Table I). The outstanding performance of AQ-PF$_6$-IL/SPE could be accredited to the integration of IL into the AQ structure (AQ-PF$_6$-IL), which enhanced the electrochemical properties by improving the electron transfer process, thereby improving the catalytic current and reducing the detection potential for the electrochemical reduction of H$_2$O$_2$.

Table I. Comparison of the AQ-PF$_6$-IL/SPE performance with other H$_2$O$_2$ sensors.

| Sensors                      | Linear range (μM) | LOD (μM) | Sensitivity (μA μM$^{-1}$ cm$^{-2}$) | References |
|------------------------------|-------------------|----------|--------------------------------------|------------|
| Fe-Cm                         | 0.56–785          | 0.19     | 0.128                                | 43         |
| Fe-S Ns/NS                    | 10–3300           | 7.9      | 4.480                                | 44         |
| Ag NW array                   | 100–3100          | 29.2     | 0.026                                | 45         |
| Fca-[BMIM][BF$_4$]-CA-SPGE    | 1–1200            | 0.35     | 0.0106                               | 46         |
| AgNP@GNR/SPCE                 | 500–5000          | 20       | 1.270                                | 47         |
| PFECS/GO/GCE                  | 10–190            | 1.2      | 0.117                                | 48         |
| Azu-A/CHO-IL/ERGO/SPE         | 30–1000           | 11.5     | 0.133                                | 49         |
| Ti/CdO/ITO                    | 10–190            | 4        | 0.270                                | 50         |
| AQ-PF$_6$-IL/SPE              | 10–1228           | 2.87     | 0.281                                | This work  |
AQ-PF₆-IL/SPE demonstrated enhanced peak currents along with a reduced formal potential compared to AQ/SPE. Further, the electrocatalytic properties on the developed sensor were explored and the fabricated sensor showed an excellent electrocatalytic reduction of H₂O₂ over a broader linear concentration range from 10⁻¹² to 12.28 μM with excellent sensitivity and limit of detection of 0.281 μA μM⁻¹ cm⁻² and 2.87 μM, respectively. Furthermore, the developed AQ-PF₆-IL/SPE sensor showed good anti-interference ability, excellent cyclic and storage stability, along with remarkable electrode reproducibility. These superior properties of the developed sensor could be attributed to the tactical design of the new redox ionic liquid hybrid molecule in which the conductivity enhancer (IL) was directly tagged to the redox moiety (AQ) itself, which facilitated the dual role (mediating and conducting) to be effectively harnessed by a single molecule. The analytical performance of the sensor is very impressive and comparable with the present day technologies and thus it can be suitably engineered and made versatile for real time detection of H₂O₂. Furthermore, the present investigation opens a new era in the design and synthesis of hybrid molecules with multiple roles for task-specific electrochemical applications.

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**Figure 5.** (A) Amperometry of AQ-PF₆-IL/SPE for the determination of H₂O₂ (0.15 mM) along with 5-fold concentrations of interferents and (B) Corresponding current response columnar diagram. (C) CVs of AQ-PF₆-IL/SPE for 50 continuous scans (Inset: Day-to-day current response) (D) Columnar diagram of current response obtained for the five identically fabricated AQ-PF₆-IL/SPE sensor in the absence and presence of 0.3 mM H₂O₂.

**Table II.** Determination of H₂O₂ spiked in milk and apple juice samples.

| Sample    | Spiked (μM) | Found (μM) | Recovery (%) | %RSD a) |
|-----------|-------------|------------|--------------|---------|
| Milk      | 150         | 144.82     | 96.54        | 2.54    |
|           | 250         | 253.61     | 101.44       | 3.62    |
| Apple Juice | 200         | 197.73     | 98.86        | 2.14    |
|           | 300         | 306.37     | 102.12       | 3.22    |

a) Triplicates were performed.
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