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The risk of mixing dilute hydrogen peroxide and acetone solutions

The present study documents the results of a literature search and experimental work to assess the risks of mixing dilute H$_2$O$_2$ and acetone solutions. The use of dilute H$_2$O$_2$ to clean chemical vessels is common, but it has been shown to be potentially hazardous due to the reaction of H$_2$O$_2$ with organic solvents to form explosive peroxides. Mixing concentrated H$_2$O$_2$ and acetone with an acid catalyst is known to form the shock and friction sensitive explosives triacetone triperoxide (TATP) and diacetone diperoxide (DADP). A search of the chemical literature was unable to identify any directly applicable research or technical information that addressed the potential formation of explosive peroxides when mixing dilute H$_2$O$_2$ and acetone solutions. The conclusion of these experiments is that when mixing dilute solutions, such as less than 3% H$_2$O$_2$ and 7% acetone, the solutions are unlikely to form significant amounts of TATP or DADP. In the presence of an acid catalyst, hundreds of parts per million of organic peroxides can be formed. Although TATP is relatively insoluble in water, it is soluble at roughly the 15 ppm level and higher for acetone and H$_2$O$_2$ solutions, thus any acetone peroxides that are formed without an acid catalyst should remain soluble in the aqueous cleaning solution.

By Jimmie C. Oxley*, Joseph Brady, Steven A. Wilson, James L. Smith

INTRODUCTION

Hydrogen peroxide mixed with organic solvents is known to form dangerous peroxides. Hydrogen peroxide and acetone is an especially hazardous combination that can form various explosive peroxides when mixed at high concentration while using an acid catalyst. The primary explosive triacetone triperoxide (TATP) is a noteworthy reaction product, because it is insoluble in water and is highly sensitive to friction, shock and temperature. It is called a primary explosive due to its ease of detonation. Note that other peroxide molecules can also be formed, including diacetone diperoxide (DADP). Figure 1 provides the molecular structures and physical properties of TATP, DADP and other peroxide products that are formed according to the literature.

The potential for forming explosive peroxides when mixing dilute solutions of H$_2$O$_2$ and acetone has not been documented. Industrial scrubbers are routinely used in chemical plants. Periodic cleaning to remove bio-sludge buildup inside the scrubber is required. One approach is the introduction of dilute aqueous hydrogen peroxide (H$_2$O$_2$). A concern is that in a scrubber used to remove acetone from a process, air might produce explosive peroxides from the reaction of H$_2$O$_2$ and acetone. To evaluate this concern, these reagents were combined in acidic media and alkaline media, and in the presence of steel coupons containing welding materials.

Prior to experimental work, a comprehensive literature search was conducted. Wolfenstein first reported the formation of TATP in 1885 when mixing equal parts of acetone with 10% H$_2$O$_2$. He found that TATP crystals were insoluble in water, acids and alkalis but readily soluble in organic solvents such as acetone. Shanley and Greenspan prepared a review article on the physical and chemical properties of highly concentrated H$_2$O$_2$ solutions in 1947. They provided a ternary diagram mapping the detonable compositions of mixtures of acetone-water-H$_2$O$_2$ solutions. The explosive power was measured by the inspection of lead pipes containing 10 mL mixtures initiated by blasting caps. There was no identification or isolation of TATP in this study. Milas and Golubovic studied the products of the reaction of H$_2$O$_2$ and acetone with and without acid. They found that solutions of acetone and hydrogen peroxide in the presence of acid would predominantly form the trimer TATP. In the absence of acid, 2,2-dihydroperoxypropane was formed and slowly converted to [dioxysis(1-methylheptylidene)]bis-hydroperoxide. Sauer and Edwards*...
Figure 1. Reaction Products of Hydrogen Peroxide and Acetone.

studied the formation of 2,2-dihydroperoxypropane at various pH values. The work provided reaction kinetics parameters, but there was no mention of TATP or DADP. They later studied the formation of higher adducts, but only proposed that TATP is formed by a reaction between acetone and [dioxobis(1-methylethylidene)]bis-hydroperoxide. Evans et al. described, in a forensic science case study, how easily TATP is formed by mixing acetone, 6% H₂O₂ and concentrated sulfuric acid. A review by Mackenzie addressed the handling and use of H₂O₂, and stressed safety precautions necessary at various strengths. There was no mention of TATP, but he did warn against the buildup of gases, including oxygen, as H₂O₂ degraded. In addition, there were warnings that after the reaction one should be cautious during process steps, such as distillation, that would concentrate any unstable peroxide compounds. A 1991 review by Mackenzie provided safety considerations for process designs that use H₂O₂ and organic chemicals. It highlighted various risks, including the dangers from decomposition of H₂O₂, as well as a variety of production hazards. U.S. patent 5,003,109 describes a process of destroying acetone peroxides formed during phenol synthesis by heating, decreasing pH, and adding a copper compound. The work implies that the chemical destruction of acetone peroxides would require conditions difficult to achieve during industrial processing. In 1999, Bellamy found that one could chemically destroy TATP by refluxing in ethanol containing tin(II) chloride. They also report that TATP is almost as powerful as trinitrotoluene (TNT), but is extremely sensitive to friction and impact sensitivity tests. TATP is quite soluble in chloroform (42.7 wt%), toluene (28.6 wt%), acetone (17.2 wt%), and hexane (14.4 wt%). Jiang et al. described the preparation of tetrameric acetone peroxide (CAS #74515-93-8). They claim that SnCl₂·5H₂O or SnCl₂·2H₂O could act as a catalyst to form a tetramer. Their results indicate that no reaction takes place when treating acetone with 30% H₂O₂ and no catalyst, but when 0.5 mmol of catalyst was added, a 19% yield of tetrameric acetone peroxide was formed. In 2002, Oxley et al. studied the decomposition of TATP and concluded that the predominant decomposition product in the gas phase is methyl acetate. In a condensed-phase, or within proton-donating solvents, TATP breaks down predominately into acetone and water. Widmer et al. developed an LC/MS method to replace GC/MS methods, stating TATP degrades GC columns. This paper also observed that TATP has two structural isomers, both stable at room temperature. Their method was sensitive to 0.1 ng per 100 μL (or 0.1 ppm). Schulte-Ladbeck developed a
trace analysis method for TATP. The method uses liquid chromatography to separate the analytes followed by post-column UV irradiation and derivatization-based fluorescence detection. Denekamp et al. further described the two structural isomers of TATP and explained that a high transition energy step was the cause of the two conformations being stable at room temperature. In 2004, Oxley et al. developed procedures for training dogs to detect TATP. The work highlights that loss of TATP from surfaces is due to its high rate of sublimation. In 2009, Oxley et al. studied the vapor density of TATP. This work determines vapor pressures of both DADP and TATP (17.7 and 7.8 Pa at 25 °C). Dubnickova et al. studied the decomposition of TATP and determined that it is not an enthalpy-based explosion, but rather, an entropic burst in which four gas molecules are created from one TATP molecule. Eyler explored the solvent effects on the thermal decomposition of TATP (135–170 °C) and found that the rate was positively influenced by solvent polarity.

**EXPERIMENTAL**

**General Experimental Set-Up**

A “mix” was defined as a combination of hydrogen peroxide (initially 50 wt%) and acetone (initially 100%) added to water to give hydrogen peroxide concentrations that varied from 30 wt% to 12 wt% to 3 wt% and the acetone concentrations of 50 wt% or 7 wt%. Catalysts included various concentrations of sulfuric acid, aqueous sodium hydroxide (30 wt%) or metal coupons. The ability of the “mix” to form TATP was judged by gas chromatography (GC) analysis equipped with either a mass selective detector (MSD) or an electron capture detector (ECD). If, after 48 hours, white solid precipitate was observed, the solids were collected, dried and analyzed by GC/MS to confirm their identity. If, after 48 hours, no solid had formed, the solutions were left for an additional 3 to 6 days and extracted with chloroform or dichloromethane, and the organic extract was analyzed by gas chromatography.

An Agilent 6890 gas chromatograph coupled with an Agilent 5973i Mass selective detector (GC/MS), and an Agilent 6890 gas chromatograph coupled with a micro-electron capture detector (GC/μECD), were employed to separate, identify and quantify TATP. The GC/MS was fitted with a 6-meter Agilent DB-5MS capillary column with a nominal diameter of 250 μm and film thickness of 0.25 μm. The carrier gas was UHP helium at an initial flow rate of 2.5 mL/min and an average linear velocity 129 cm/sec. The inlet was run with a 5:1 split at 170 °C at a pressure of 1.09 psi. The oven was held at 50 °C for 2 minutes, ramped to 190 °C at 20 °C per minute and the transfer line to the MSD was held at 275 °C. To ensure that all materials had passed through the column, a 1 minute post run at 280 °C with a flow rate of 8 mL/minute was used.

The GC/μECD was fitted with a 6-meter Restek RTX-200 capillary column with a nominal diameter of 530 μm and a film thickness of 1.50 μm. The carrier gas was UHP helium at a rate of 15.8 mL/minute with an average linear velocity of 117 cm/sec. The inlet was held at 170 °C in the splitless mode for 0.50 minutes before the inlet purged at 40.0 mL/minute. The makeup gas to the μECD was UHP nitrogen run in the constant flow mode at a rate of 30 mL/minute and the detector was held at 280 °C. The oven was initially held at 40 °C for 2 minutes, ramped at 20 °C/minute to 150 °C and held for 1 minute, followed by a 0.50 minutes post run at 250 °C.

The presence and quantity of TATP was determined by matching the retention times and mass spectra of samples to authentic standard solutions. A calibration curve for TATP was developed by dissolving a known amount of authentic TATP in dichloromethane and preparing concentrations that bracketed the amounts that were expected during an analytical run. A typical set of concentrations for an analytical run on the GC/MS was 100, 200, 300, 400 and 500 mg/L of TATP. A typical set of concentrations for an analytical run on the GC/μECD was 0.5, 1, 2.5, 5, 10, 25, and 50 mg/L. A linear dependence was determined by measuring the peak height or integrating the area under the characteristic peak for TATP and plotting the area or height versus concentration (Figure 2).

![Figure 2. Calibration Curve for TATP by GC/μECD.](image)

It should be noted that TATP chromatograms exhibit two peaks, one for each conformational isomer. Under the GC conditions used in Figure 3, the isomeric peaks eluted ~9 minutes apart. When each peak was examined by GC/MS, the mass spectra were identical, confirming that the two peaks are isomers as reported by Denekamp et al. (Figure 3).

**Solubility of TATP in Acetone and Aqueous Acetone Solutions**

The solubilities of TATP and DADP in pure acetone were measured as 151 mg/mL (0.191 mg/mg) and 45 mg/mL (0.0548 mg/mg) respectively. TATP was found to have a solubility of 200 mg/mL (0.298 mg/mg) in chloroform. These values were determined by adding solvent drop wise to the solid peroxide while stirring in a round-bottom flask. Qualitative solubility tests in aqueous acetone and
aqueous hydrogen peroxide are summarized in Table 1. Solubility was determined by visual inspection.

**Mixing under Acidic Conditions**

Solutions for the acidic media conditions were prepared as follows; the water necessary for dilution was placed in an Erlenmeyer flask, and the flask was immersed in an ice bath. Hydrogen peroxide (0.10 mol) and then acetone (0.10 mol) were added to the chilled water in proportions such that the final concentration of the mixture mimicked the combination of 30%, 12%, or 5% (w/w) hydrogen peroxide with 50% or 7% (w/w) acetone. Each of the six possible combinations was prepared in triplicate and either 0, 1 or 5 drops of concentrated sulfuric acid was added to each of the samples. The average mass of one drop of concentrated sulfuric acid was found to be 50 mg.

The flasks were sealed with Parafilm and left at room temperature for 2 to 7 days. If a precipitate formed after 2 days, it was removed by filtration, washed with water, dissolved in acetonitrile, and analyzed by GC/MS. If no precipitate was observed after 7 days, a 10 mL aliquot of the aqueous solution was extracted twice with 10 mL of dichloromethane, the organic extracts were combined, rinsed twice with 10 mL of deionized water, dried over magnesium sulfate and that solution was analyzed by GC/MS (Tables 2 and 3).

**Mixing with Welded Steel Coupons**

Three types of steel coupons were evaluated to determine the catalytic effects associated with formation of TATP. The three types were a coupon without a weld, a coupon with a “dirty” weld, and a coupon with a “clean” weld. A single coupon of each type was placed in the six acetone/hydrogen peroxide mixtures. The reaction vessels were sealed and left at room temperatures for approximately 100 hours. An aliquot was removed and analyzed by GC/MS as described above. Only the 50%/30% and 50%/12% mixtures of hydrogen peroxide/acetone indicated the presence of TATP by GC/MS. Table 4 shows the configurations that were examined and the concentration of TATP observed in these solutions.

**Alkaline Conditions**

The most concentrated mixture investigated in the presence of acid was re-investigated in the presence of base (50% acetone/30% hydrogen peroxide). In a 125 mL Erlenmeyer flask, water, hydrogen peroxide and acetone were combined so that acetone was 50 wt% and hydrogen peroxide was 30 wt%. Drops of sodium hydroxide solution, 0, 1, 2, or 10, were added.

![Figure 3. Chromatograms of Authentic TATP Standard Solutions. From Top to Bottom: 50, 25, 10, 5, 2.5, 1 mg/L TATP Standard Solutions.](image)

---

**Table 1. Solubility of TATP in Various Solutions.**

<table>
<thead>
<tr>
<th>TATP (mg)</th>
<th>H₂O Amount (kg)</th>
<th>Acetone (ppm)</th>
<th>H₂O₂ (g)</th>
<th>H₂O₂ (wt%)</th>
<th>Dissolved</th>
<th>Not dissolved</th>
<th>TATP (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>24.7</td>
<td>1.8000</td>
<td></td>
<td></td>
<td></td>
<td>100 hours</td>
<td>13.7</td>
<td></td>
</tr>
<tr>
<td>25.6</td>
<td>1.6000</td>
<td></td>
<td></td>
<td></td>
<td>100 hours</td>
<td>16</td>
<td></td>
</tr>
<tr>
<td>24.5</td>
<td>1.0000</td>
<td>100</td>
<td></td>
<td></td>
<td>100 hours</td>
<td>24.5</td>
<td></td>
</tr>
<tr>
<td>25.3</td>
<td>0.9999</td>
<td>100</td>
<td></td>
<td></td>
<td>100 hours</td>
<td>25.3</td>
<td></td>
</tr>
<tr>
<td>51</td>
<td>2.0001</td>
<td>100</td>
<td></td>
<td></td>
<td>72 hours</td>
<td>25.5</td>
<td></td>
</tr>
<tr>
<td>25.4</td>
<td>1.0001</td>
<td>150</td>
<td></td>
<td></td>
<td>72 hours</td>
<td>25.4</td>
<td></td>
</tr>
<tr>
<td>24.2</td>
<td>1.0000</td>
<td>150</td>
<td></td>
<td></td>
<td>72 hours</td>
<td>24.2</td>
<td></td>
</tr>
<tr>
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<td>0.2504</td>
<td>799</td>
<td></td>
<td></td>
<td>72 hours</td>
<td>101</td>
<td></td>
</tr>
<tr>
<td>24.8</td>
<td>1.0002</td>
<td>200</td>
<td></td>
<td></td>
<td>72 hours</td>
<td>24.8</td>
<td></td>
</tr>
<tr>
<td>50.2</td>
<td>0.9999</td>
<td>130</td>
<td></td>
<td></td>
<td>96 hours</td>
<td>50.2</td>
<td></td>
</tr>
<tr>
<td>75.8</td>
<td>1.9999</td>
<td>105</td>
<td></td>
<td></td>
<td>96 hours</td>
<td>37.9</td>
<td></td>
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<td>25.6</td>
<td>1.0316</td>
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<td></td>
<td></td>
<td>96 hours</td>
<td>24.8</td>
<td></td>
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<tr>
<td>25</td>
<td>0.4737</td>
<td>0.585</td>
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<td>96 hours</td>
<td>52.8</td>
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<td>12%</td>
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<td>24 hours</td>
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<td>24.8</td>
<td>0.5000</td>
<td>50</td>
<td>12%</td>
<td></td>
<td>96 hours</td>
<td>49.6</td>
<td></td>
</tr>
</tbody>
</table>

---

Each solution was prepared in duplicate. The flasks were sealed and left undisturbed at room temperature for 8 days. The mixtures were twice extracted as described above and the extracts were analyzed for TATP by GC/µECD. The concentration of the NaOH solution was determined by recording the mass necessary to fill a 2 mL volumetric flask and repeated three times. The density was found to be 1.522 g/mL. By comparing this value to literature values, the solution was found to be 48.58% NaOH by mass. The mass of a drop of the NaOH was determined to be 38 mg by recording the mass of 10 individual drops and taking the average. After 8 days, GC–µECD analysis of the organic extracts of the alkaline solutions showed no detectable TATP had been generated, while two control solutions (acetone, hydrogen peroxide, no NaOH) were found to contain TATP in excess of 50 mg/L.

### Examination of Dilute Acids in Acetone/Peroxide Mixture

To determine if more dilute solutions of acetone in hydrogen peroxide could form TATP, solutions of acetone (7% and 0.04%) were mixed with hydrogen peroxide solutions (12% and 3%). The 7% acetone solutions were combined with the hydrogen peroxide solutions such that the mixture contained 0.1 mol of each reactant, theoretically yielding 0.053 mol TATP. The 0.04% acetone solutions were combined with hydrogen peroxide such that each mixture contained 0.01 mol of each reactant, theoretically yielding 0.0053 mol TATP. After acetone and hydrogen peroxide were combined, one drop of sulfuric acid (18 M, 13 M, 5 M, or 0.1 M) was added, and the mixtures were sealed and left at room temperature for ~75 hours. The mixtures from the 12% and 7% acetone were extracted twice with 20 mL dichloromethane twice. The organic extracts were combined and rinsed with 10 mL of water to remove any residual acid or acetone and dried over magnesium sulfate. The mixtures from the 0.04% acetone solutions had a total volume of approximately 1,560 mL and were extracted twice with 50 mL dichloromethane and rinsed once with 20 mL water and dried over magnesium sulfate (Table 5).

### RESULTS AND DISCUSSION

#### Solubility of TATP in Water

TATP is practically insoluble in water, but is soluble in acetone. Solubility values of dilute aqueous solutions of acetone have not been published. In this study, 25 mg of TATP was added to various aqueous solutions of acetone

---

**Table 2. Mixes Prepared to Test TATP Formation Under Acidic Conditions.**

<table>
<thead>
<tr>
<th>TATP ppt in 48 hours</th>
<th>Acetone (%)</th>
<th>H₂O₂ (%)</th>
<th>H₂SO₄ (d.)</th>
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</thead>
<tbody>
<tr>
<td>50</td>
<td>30</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>50</td>
<td>12</td>
<td>5</td>
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<td>50</td>
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<td></td>
</tr>
<tr>
<td>50</td>
<td>12</td>
<td>1</td>
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</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>TATP in solution after 7 days</th>
<th>Acetone (%)</th>
<th>H₂O₂ (%)</th>
<th>H₂SO₄ (d.)</th>
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</thead>
<tbody>
<tr>
<td>50</td>
<td>3</td>
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<td>3</td>
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<table>
<thead>
<tr>
<th>No TATP after 7 days</th>
<th>Acetone (%)</th>
<th>H₂O₂ (%)</th>
<th>H₂SO₄ (d.)</th>
</tr>
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<tbody>
<tr>
<td>50</td>
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<tr>
<td>7</td>
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**Table 3. TATP Found in Solutions Without Precipitate after 7 Days.**

<table>
<thead>
<tr>
<th>Acetone</th>
<th>Peroxide</th>
<th>Acid (drops)</th>
<th>Mass % Acetone</th>
<th>Mass % Peroxide</th>
<th>Mass % Reactants</th>
<th>Mass TATP in solution (µg)</th>
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<tbody>
<tr>
<td>7%</td>
<td>30%</td>
<td>5</td>
<td>6.2%</td>
<td>3.6%</td>
<td>9.8%</td>
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<td>12%</td>
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<td>3.1%</td>
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<td>5.2%</td>
<td>2.7%</td>
<td>8.3%</td>
<td>Not found</td>
</tr>
<tr>
<td>7%</td>
<td>3%</td>
<td>0</td>
<td>3.0%</td>
<td>1.7%</td>
<td>4.7%</td>
<td>Not found</td>
</tr>
</tbody>
</table>
Table 4. TATP Found in Solutions with Steel Coupons.

<table>
<thead>
<tr>
<th>Acetone, Initial conc.</th>
<th>HP, Initial conc.</th>
<th>Weld type</th>
<th>Mass % Acetone</th>
<th>Mass % Peroxide</th>
<th>Mass % Reactants</th>
<th>Mass TATP (µg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50%</td>
<td>30%</td>
<td>C</td>
<td>25.3%</td>
<td>14.9%</td>
<td>40.2%</td>
<td>22,636</td>
</tr>
<tr>
<td>50%</td>
<td>30%</td>
<td>D</td>
<td>25.2%</td>
<td>15.0%</td>
<td>40.2%</td>
<td>26,536</td>
</tr>
<tr>
<td>50%</td>
<td>30%</td>
<td>N</td>
<td>25.3%</td>
<td>14.8%</td>
<td>40.1%</td>
<td>23,770</td>
</tr>
<tr>
<td>50%</td>
<td>12%</td>
<td>C</td>
<td>14.6%</td>
<td>8.5%</td>
<td>23.1%</td>
<td>4,947</td>
</tr>
<tr>
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<td>12%</td>
<td>D</td>
<td>14.6%</td>
<td>8.6%</td>
<td>23.1%</td>
<td>4,153</td>
</tr>
<tr>
<td>50%</td>
<td>12%</td>
<td>N</td>
<td>14.4%</td>
<td>8.4%</td>
<td>22.8%</td>
<td>4,481</td>
</tr>
<tr>
<td>50%</td>
<td>3%</td>
<td>C</td>
<td>4.7%</td>
<td>2.7%</td>
<td>7.4%</td>
<td>0</td>
</tr>
<tr>
<td>50%</td>
<td>3%</td>
<td>D</td>
<td>4.6%</td>
<td>2.7%</td>
<td>7.3%</td>
<td>0</td>
</tr>
<tr>
<td>50%</td>
<td>3%</td>
<td>N</td>
<td>4.7%</td>
<td>2.7%</td>
<td>7.4%</td>
<td>0</td>
</tr>
<tr>
<td>7%</td>
<td>30%</td>
<td>C</td>
<td>6.1%</td>
<td>3.6%</td>
<td>9.8%</td>
<td>0</td>
</tr>
<tr>
<td>7%</td>
<td>30%</td>
<td>N</td>
<td>6.2%</td>
<td>3.6%</td>
<td>9.8%</td>
<td>0</td>
</tr>
<tr>
<td>7%</td>
<td>12%</td>
<td>N</td>
<td>5.2%</td>
<td>3.1%</td>
<td>8.3%</td>
<td>0</td>
</tr>
<tr>
<td>7%</td>
<td>12%</td>
<td>D</td>
<td>5.2%</td>
<td>3.1%</td>
<td>8.3%</td>
<td>0</td>
</tr>
<tr>
<td>7%</td>
<td>3%</td>
<td>C</td>
<td>3.0%</td>
<td>1.7%</td>
<td>4.7%</td>
<td>0</td>
</tr>
<tr>
<td>7%</td>
<td>3%</td>
<td>D</td>
<td>3.0%</td>
<td>1.7%</td>
<td>4.7%</td>
<td>0</td>
</tr>
<tr>
<td>7%</td>
<td>3%</td>
<td>N</td>
<td>3.0%</td>
<td>1.7%</td>
<td>4.7%</td>
<td>0</td>
</tr>
</tbody>
</table>

Weld type: C = cleaned weld, D = dirty weld, N = no weld.

Table 5. TATP Found in Solutions Prepared with Dilute Acid.

<table>
<thead>
<tr>
<th>Initial Acetone</th>
<th>Initial H₂O₂ Conc.</th>
<th>Acid Conc.</th>
<th>Mass TATP (µg)</th>
<th>pH</th>
<th>Mass % Acetone</th>
<th>Mass % H₂O₂</th>
<th>Mass % Reactants</th>
</tr>
</thead>
<tbody>
<tr>
<td>7%</td>
<td>12%</td>
<td>18 M</td>
<td>4,993</td>
<td>2.41</td>
<td>5.27%</td>
<td>3.06%</td>
<td>8.33%</td>
</tr>
<tr>
<td>7%</td>
<td>12%</td>
<td>18 M</td>
<td>2,669</td>
<td>2.64</td>
<td>5.21%</td>
<td>3.10%</td>
<td>8.32%</td>
</tr>
<tr>
<td>7%</td>
<td>12%</td>
<td>13 M</td>
<td>2,803</td>
<td>2.65</td>
<td>5.22%</td>
<td>3.07%</td>
<td>8.29%</td>
</tr>
<tr>
<td>7%</td>
<td>12%</td>
<td>5 M</td>
<td>1,632</td>
<td>2.86</td>
<td>5.21%</td>
<td>3.09%</td>
<td>8.50%</td>
</tr>
<tr>
<td>7%</td>
<td>5%</td>
<td>18 M</td>
<td>279</td>
<td>2.86</td>
<td>2.96%</td>
<td>1.74%</td>
<td>4.70%</td>
</tr>
<tr>
<td>7%</td>
<td>5%</td>
<td>18 M</td>
<td>170</td>
<td>2.63</td>
<td>2.97%</td>
<td>1.73%</td>
<td>4.70%</td>
</tr>
<tr>
<td>7%</td>
<td>5%</td>
<td>13 M</td>
<td>154</td>
<td>2.76</td>
<td>3.13%</td>
<td>1.74%</td>
<td>4.87%</td>
</tr>
<tr>
<td>7%</td>
<td>5%</td>
<td>5 M</td>
<td>103</td>
<td>2.98</td>
<td>2.96%</td>
<td>1.73%</td>
<td>4.68%</td>
</tr>
<tr>
<td>7%</td>
<td>5%</td>
<td>0.1 M</td>
<td>0</td>
<td>3.11</td>
<td>2.99%</td>
<td>1.73%</td>
<td>4.72%</td>
</tr>
<tr>
<td>0.04%</td>
<td>5%</td>
<td>18 M</td>
<td>0</td>
<td>–</td>
<td>0.04%</td>
<td>0.02%</td>
<td>0.06%</td>
</tr>
<tr>
<td>0.04%</td>
<td>5%</td>
<td>18 M</td>
<td>0</td>
<td>–</td>
<td>0.04%</td>
<td>0.02%</td>
<td>0.06%</td>
</tr>
<tr>
<td>0.04%</td>
<td>5%</td>
<td>15 M</td>
<td>0</td>
<td>–</td>
<td>0.04%</td>
<td>0.02%</td>
<td>0.06%</td>
</tr>
<tr>
<td>0.04%</td>
<td>5%</td>
<td>5 M</td>
<td>0</td>
<td>–</td>
<td>0.04%</td>
<td>0.02%</td>
<td>0.06%</td>
</tr>
<tr>
<td>0.04%</td>
<td>5%</td>
<td>0.1 M</td>
<td>0</td>
<td>–</td>
<td>0.04%</td>
<td>0.02%</td>
<td>0.06%</td>
</tr>
<tr>
<td>7%</td>
<td>12%</td>
<td>–</td>
<td>–</td>
<td>3.84</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>–</td>
<td>50%</td>
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<td>–</td>
<td>1.24</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>–</td>
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<td>–</td>
<td>–</td>
<td>3.44</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

and visually inspected to determine if the solid dissolved. Table 1 is a compilation of the results. The TATP solubility in neat water was determined to be below ~15 mg/L while solubility in a 0.1% acetone solution exceeded 25 mg/L for TATP. TATP solubility in the two H₂O₂ solutions screened was found to be greater than 50 mg/L. The slight solubility of TATP in water is slightly increased by the inclusion of acetone and H₂O₂.

Influence of Concentration and Acid Catalyst on TATP Formation

This experiment was conducted to determine if TATP would be formed at concentrations that might be encountered in an industrial process. Table 2 lists the results of this experiment. Note that the concentrations of H₂O₂ were initially 30%, 12%, and 3%, while the concentrations of acetone were initially 50% and 7%. The higher concentrations were included to provide levels known to make TATP with the purpose of verifying the experimental methods of observing or measuring TATP. Solid TATP was seen in the highest concentrations with 50% acetone and 30% or 12% H₂O₂, with the sulfuric acid catalyst, and small amounts of precipitate were observed in the 50% acetone, 30% H₂O₂ mixture. After 7 days, the remaining solutions were extracted and analyzed by GC/µECD. TATP was detected in all
solutions in which sulfuric acid catalyst had been added. Note that for the solutions with no acid, TATP was seen only in the 50% acetone and 30% H$_2$O$_2$ solution; thus, the presence of an acid catalyst appears to be a key ingredient for making TATP in dilute solutions.

**Influence of 304L Stainless Steel and Welds on TATP Formation**

To evaluate if stainless steel surfaces or stainless steel welds might catalyze the formation of TATP, coupons were obtained to serve as examples of the 304L stainless steel metal commonly used to build an industrial scrubber. Coupons were also obtained which represented clean and dirty welds on 304L stainless steel to evaluate the influence of weld seams. The solutions from the above experiments, with no acid catalyst present (Table 2), were re-evaluated using the metal coupons as potential catalyst materials. The results are shown in Table 4. TATP was detected only in the 50% acetone and 50% H$_2$O$_2$ solution at nearly identical concentrations to those without the coupons. This experiment suggests that 304L stainless steel does not catalytically influence the formation of TATP.

**Influence of Acidity on TATP Formation**

The formation of TATP is an acid catalyzed reaction, but the amount of acid required to generate the peroxide has not been published. Solution acidity is an important consideration for establishing a safe boundary for the use of H$_2$O$_2$ in the presence of acetone. H$_2$O$_2$ solutions are often sold and stored under acidic conditions for increased stability. Table 5 contains the results of an evaluation of the influence of acidity. No TATP was detected at any acid level for the 0.04% acetone solutions. There was also no TATP detected for the 7% acetone and 3% H$_2$O$_2$ with 0.1 M acid solution added. These results highlight the need for sufficient acid concentration in forming TATP.

**Alkaline Conditions**

The literature concerning the formation of TATP focuses on the use of acid catalysts and there is no mention on the effects of alkalinity. To evaluate these conditions, test solutions were prepared with three different concentrations of NaOH. No TATP was detected in these experiments, indicating that the formation of TATP is not base catalyzed and, furthermore, that the presence of trace amounts of NaOH inhibits the formation of TATP.

**CONCLUSIONS**

Oxidation of acetone with hydrogen peroxide produces TATP. While this reaction is relatively gentle, the product, TATP, is highly sensitive to shock and friction that results in explosive decomposition. Situations where TATP accumulation are highly hazardous. This study was initiated to assess the possibility that TATP accumulation could result from cleaning processes for industrial scrubbers which use dilute hydrogen peroxide and acetone. Hydrogen peroxide solutions are mildly acidic and even solutions containing only a few percent of acetone and hydrogen peroxide can form TATP. Without additional catalytic activity, the formation of small quantities of TATP in dilute H$_2$O$_2$ and acetone in aqueous solutions is likely to remain soluble in the reaction media. The presence of 304L stainless steel did not catalyze the formation of TATP. The presence of trace amounts of sodium hydroxide in solutions that normally form TATP inhibits the formation of TATP in all the configurations screened here. The cyclic dimer, DADP, was not found in any of the configurations, leading one to conclude that the formation of DADP necessitates a relatively high concentration of acid catalyst. Acid catalyzed mixtures of acetone and hydrogen peroxide is capable of producing hazardous amounts organic peroxides (i.e., hundreds of ppm) which could accumulate. This study shows that the simple mixing of dilute solutions of hydrogen peroxide and acetone does not appear to accumulate TATP in hazardous amounts. The solubility of TATP in pure water is about 15 ppm and somewhat higher when acetone and peroxide are present. The small amount of TATP produced without acid catalysis is likely to remain soluble in the aqueous cleaning solution.

**REFERENCES**

1. Wolfenstein, R. Ber. 1885, 28(1459), 2265.