Forensic Accident Investigation Using Pyrotechnic Reaction Residue Particle Analysis

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ABSTRACT

Pyrotechnic reaction residue particle (PRRP) production, sampling and their basic analyses are similar to that for primer gunshot residue (PGSR). Both types of particles originate from energetic chemical reactions that generate products that are initially melted and/or vaporized. These chemical reaction products are dispersed by the temporary and permanent gases from the chemical reaction. Then the reaction products solidify and are deposited as tiny spheroidal particles on objects in the area. Sampling is accomplished using conductive carbon adhesive dots. Analysis is performed using scanning electron microscopy to locate potential PRRPs and energy dispersive X-ray spectroscopy is used to characterize the signature of each suspect particle’s constituent chemical elements.

Often, standard micro-analytical chemistry performed on pyrotechnic residues cannot be expected to provide sufficient information for an investigation of the cause and course of an accident. On those occasions, PRRP analysis generally provides important information that is not otherwise available. For example, there are times when standard micro-analytical chemistry will fail to discriminate sufficiently between materials of pyrotechnic origin and other unrelated substances also present on the items being sampled. In addition, there are times when PRRP analysis can help identify details concerning the cause and course of events that are simply beyond the capability of micro-analytical chemistry.

Introduction

In the course of pyrotechnic reactions, residues (reaction products) are generated. This is true whether the reaction is occurring in the primer of a small arms ammunition cartridge, in a pipe bomb or in a firework device. A large portion of the reaction products from nearly all pyrotechnic compositions are solids at room temperature. As such these reaction products are potentially available for collection and analysis using a methodology similar to that used for primer gunshot residues (PGSR). In many cases, pyrotechnic reaction residue particle (PRRP) analysis is a useful adjunct to conventional micro-analytical chemistry since it can provide forensic information not otherwise provided (or not provided with a high confidence level).

Much of the current interest in pyrotechnic reaction residue particle (PRRP) analysis [1–4] originated with articles expressing concern that some fireworks might be capable of producing residue particles meeting the forensic criteria for PGSR [5–7]. PRRP production, sampling and their basic analysis are similar to that of PGSR. Both types of residue originate from energetic chemical reactions that generate products that are initially melted and/or vaporized. These chemical reaction products are dispersed by the temporary and permanent gases produced by the reaction. Then the reaction products solidify as tiny spheroidal particles deposited on objects in the area.

Residue sampling is generally accomplished using conductive carbon adhesive dots or tape. Then the preferred method of analysis of those residues is to use scanning electron microscopy (SEM) to locate suspect pyrotechnic reaction residue particles. Those suspect particles are then analyzed in the SEM using X-ray energy dispersive spectroscopy (EDS) to develop the suspect particle’s signature of constituent chemical elements.

As an example of when and to what extent PRRP analysis can be helpful, consider the following. Standard micro-analytical chemistry may fail to discriminate sufficiently between pyrotech-
nic residues and other unrelated substances also present on items sampled after an accident. These unrelated substances may predate the pyrotechnic incident, they may be contributed during the course of the event but are not from the pyrotechnic composition, they may be deposited post-event, or they may result from any combination of these three possibilities.

When unrelated materials are known to be present, sometimes they can be successfully accounted for with standard micro-analytical chemical analyses, although this will require additional or more complex analyses. However, when unrelated material is not known to exist or if it shares chemical species in common with the pyrotechnic composition, erroneous information is likely to result. In such cases, these unrelated residues could be incorrectly identified as being part of the pyrotechnic residue, or those species in common could be missed because of having been attributed to a non-pyrotechnic source. In this case the morphologic specificity of PRRP (spheroidal particles that range from about 0.5 to 20 micrometers) will generally allow the successful differentiation between PRRP and non-PRRP (background) materials. This plus an analysis of the chemical elements present in the particles, provide highly reliable identification of PRRP and the characteristics of the pyrotechnic material that produced them.

Another example of the special capabilities of PRRP analysis is that it can be used to differentiate between pyrotechnic residues present on the same item, but contributed by different pyrotechnic sources, possibly at different times. With such information, insight can often be gained into the cause and course of an incident that could never have been possible using standard analytical methods. This is because standard micro-analytical chemistry produces a single set of results representing the combined total of the various different pyrotechnic residues present on a sampled item, whereas PRRP analysis can generally differentiate between the different pyrotechnic residue sources.

The special capabilities of PRRP analyses are demonstrated later in this paper by reviewing three specific examples.

**SEM/EDS Basics**

In its simplest terms, the operation of a SEM can be described as follows. An electron gun pro-

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**Figure 1.** An illustration of some aspects of production and collection of secondary electrons in a SEM.

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duces high-energy electrons that are focused and precisely directed toward a target specimen in a vacuum (Figure 1). As a result of this bombardment, among other things, low energy secondary electrons are produced through interactions between the beam electrons and the atoms in the specimen. In one commonly-used SEM mode, these secondary electrons are collected and used to generate an electronic signal, where the amplitude of the signal is dependent on the nature and spacial features of the portion of the specimen being bombarded at any given time. The impinging electron beam can be systematically moved over the specimen in a rasterized pattern of scans

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**Figure 2.** An illustration of some aspects of the production and collection of secondary rasterized SEM scanning to produce an electron in a SEM image.
(Figure 2). The resulting secondary electron signal can then be used to create an overall (television-like) image of that portion of the specimen being scanned. Because the incident beam of electrons is highly focused and because the pattern of scans across the specimen can be precisely (microscopically) controlled, the image produced is of high spatial resolution and can be highly magnified (easily to 20,000 times).

In addition to the production of secondary electrons, another result of the interaction of the electron beam with the target specimen is the production of X-rays. These X-rays are uniquely characteristic of the atoms that produced them. By detecting and analyzing the energies of the X-rays that are generated, the identity of the chemical elements in the target specimen can be uniquely determined.

The most common method for analyzing the X-rays produced by the specimen is described as energy dispersive spectroscopy (EDS) using a solid state X-ray detector as shown in Figure 3.

The output of this detector consists of electronic pulses that are proportional to the X-ray energies being deposited in the detector. Using a multi-channel analyzer (MCA), the pulses are sorted according to voltage (i.e., X-ray energy) and the results (Figure 4) are then stored for subsequent interpretation (i.e., the identification of the atomic elements present).

Accordingly, the combination of SEM/EDS allows (with some limitations) the microscopic imaging of specimens and the determination of the chemical elements present in the individual particles in those specimens. This is a powerful combination of capabilities that allows for the rapid identification and characterization of PRRP.

Particle Morphology

Most often there are easily discernable differences between PRRP morphologies and those of geologic soil particles. As shown in Figure 5 the vast majority of PRRP are spherical or at least spheroidal in shape, with a substantial fraction of those particles falling in the range of 0.5 to 20 micrometers in diameter. By comparison, most soil (dirt) particles in this same size range have angular features as shown in Figure 6. However, this difference cannot be absolutely relied upon. Some geologic particles can be spheroidal as the result of their having been mobile in the environment for a long time, during which time abrasive action tends to remove their sharp angular features. Also some organic particles (e.g., grass pollen) are roughly spheroidal in shape and are in the size range of PRRP. Even though morphology alone does not provide absolute certitude with regard to identifying PRRP, using morphology allows one to eliminate the vast majority of non-PRRP from consideration. Then using EDS, the remaining non-PRRP can readily be eliminated from consideration.
Figure 5. Examples of 10 to 20 micrometer spheroidal pyrotechnic reaction residue particles (PRRP).

Figure 6. Four examples of typical 10 to 20 micrometer particles of geologic origin (soil).

Basic PRRP Methodology

The basic methodology of PRRP analysis has much in common with standard forensic trace analysis using an SEM with EDS capability. First samples are collected, generally from objects in the immediate area of a pyrotechnic event and also from persons and their clothing when that is available. In many cases the residues on objects near the seat of the event will be visible as a whitish, grayish or blackish haze on the surface of the objects. Residue samples are collected using conductive carbon adhesive dots (often 13 mm in diameter) such as commonly used for SEM work.

Except when attempting to produce relatively high quality images, the residue samples are generally not carbon or sputter coated prior to analysis. This is because the PRRP and other particles collected are generally sufficiently conductive that they do not cause serious problems with charging from the electron beam. Also, because atomic number discrimination, based on back scatter contrast differences, is rarely if ever useful in identifying PRRP, the SEM is typically operated in the secondary electron mode. Accordingly minor problems with excessive contrast can generally be tolerated.

In the search for and characterization of PRRP on an object thought to have been exposed to a pyrotechnic event, primary attention is paid to those particles of the correct morphology, as discussed above. These suspect particles are then analyzed using X-ray EDS and the information is archived. During this particle search process, usually a moderate number of non-PRRP are also investigated using EDS. This helps to establish the components present in the background particles on that specific item. (It is possible that this background material may be different than that on other items and in bulk background samples taken from the area in general.) Analysis of the background components on individual items may thus be useful to increase one's confidence in the identification and characterization of PRRP in some cases.

Having found and characterized a sufficient quantity of PRRP, it will always be possible to conclude something definitive about the nature of the pyrotechnic material involved. However, it is often necessary for the analyst to have a thorough knowledge of the pyrotechnic chemistry of both the materials used and their reaction products under the conditions of the event. This may be more complicated than might at first be appreciated, and it is sometimes necessary to use thermo-chemical modeling to correctly account for the reaction products. (While most interesting, because of its length, a proper discussion of this subject must be delayed for a subsequent paper.)

As a brief example, consider the X-ray spectra shown in Figure 7. These were produced as part of the investigation of an accident where an individual received burns when a firework allegedly exploded and sent burning pieces of pyrotechnic composition in his direction. Uppermost is the gross spectrum of the unreacted firework composition taken from the type of firework alleged to have been responsible for the injury. In the middle
is a spectrum that is typical of a PRRP produced by burning this same pyrotechnic composition under laboratory conditions. Lowermost is a spectrum that is typical of PRRPs taken from the clothing of the burn victim. In comparing the two lower spectra, note that the spectrum of PRRPs from the victim is consistent with having been produced by the suspect firework. (Note that “consistent with” is not the same as “produced by”. It is certainly possible that a number of other similar pyrotechnic devices could have been the source of the residue.)

**Figure 7**—Three X-ray spectra produced during an accident investigation.

During another investigation, one involving a night club fire, a quite different conclusion was reached. In that case, when a similar set of comparisons to those in Figure 7 was performed, it was shown that the residues from the pyrotechnic material could not have been produced by the product that was alleged. (In this case the results were found to be consistent with another product that was available to the band’s pyrotechnician.)

**Applied PRRP Methodology**

Three examples of PRRP analyses are presented to demonstrate in greater detail the unique capabilities of the method.

**Example 1: Background Discrimination**

When a pyrotechnic device explodes on the ground, the force of the explosion will mobilize a substantial amount of soil material, some of which will be deposited on remnants of the device and other objects in the area. This process, however, does not result in the mixing of the soil and pyrotechnic components within the individual particles being deposited. The magnitude of the temperature and duration of the explosion event is generally not sufficient to vaporize or even melt the soil particles. As a result, intimate co-mingling of soil and pyrotechnic components does not occur. In addition, because they have not melted, soil particles tend to maintain their generally non-spheroidal morphology. Accordingly, the normal PRRP methodology works well to differentiate successfully between PRRP and soil material. There can, however, be a complication wherein some pyrotechnic reaction products may tend to collect on the surface of the mobilized soil particles that are within the gas cloud of the explosion. This tends to occur when there is a large difference between the high temperature of an explosion fireball and the much lower condensation temperatures of some of the reaction products. The example that follows is one case where this occurred. (While this is discussed briefly below, a more complete discussion must be deferred to a future paper.)

Because of the large degree of commonality between the chemical elements present in inorganic soil components and pyrotechnic compositions combined with a somewhat similar range of solubility and reactivity, conventional microanalytical chemistry may fail to differentiate sufficiently between the two sources. Further, even a direct comparison between the samples collected from the immediate area of the explosion and PRRP-free soil may fail to produce fully definitive analytical chemical results regarding the nature of the pyrotechnic residues present. In part this difficulty results from the fact that the PRRP present on sampled items after an explosion on the ground can be overwhelmed by the larger amounts of soil collecting on those items. In that case the components present as PRRP may not be detected.

A demonstration was conducted using a small polyethylene container, a common 35-mm film canister, filled with approximately 28 g of a typical fireworks flash powder (70% potassium per-
chlorate and 30% aluminum powder). The container was placed directly on the ground and caused to explode using an electric match that had been installed in the device (Figure 8). Following the event, residues were collected from the previously cleaned surfaces of objects placed in the immediate area of the explosion. One collecting surface, as seen in Figure 8, was positioned approximately 150 mm from the explosive charge. The collecting surface was a small piece of 3-mm thick tileboard with a hard, thick and tightly laminated surface. Figure 9 presents a series of three EDS spectra, two produced by the residues on the test surfaces plus one from a sample of unaffected dirt (labeled “Pristine Dirt”).

![Figure 8. Setup for background discrimination demonstration, showing a collection surface attached to a heavy metal support and a 35-mm film canister containing fireworks flash powder.](image)

The top spectrum of Figure 9, labeled “1) "Dirt" plus PRRP” is a composite of gross EDS spectra, taken at low magnification and scanning over relatively large portions of the post explosion residue sample. This gross EDS spectrum integrates the results of a very large number of individual particles in the sample. In the spectrum the major peaks are from aluminum, silicon, sulfur, potassium, calcium and iron. When examined more closely, minor peaks from sodium, magnesium, chlorine, titanium and copper can be seen to be present as well. The second spectrum labeled as “2) Pristine Dirt” is a composite of the spectra from 16 individual soil component grains. Based on the different assemblages of the constituent elements present, the individual soil particles fell into four reasonably distinct and recognizable geologic categories that were consistent with the clay-based soil. However, this is a composite (i.e., average) spectrum of the four types of soil particles.

![Figure 9. Collection of three composite spectra taken from samples of pyrotechnic residues co-mingled with soil.](image)

The results embodied in spectra 1 and 2 are similar to what would be expected if conventional micro-analytical chemistry were performed on these two samples, with the exception that chemical species (ions) and not just the elements present might be determined (depending on the analytical method chosen). These two spectra are quite similar to each other especially considering that any two samples of pristine dirt (both without the presence of PRRP) would be expected to have slight differences in the quantities of the elements present because of the naturally variable mixture of its four primary soil components. Accordingly, while it is likely that standard micro-analytical chemistry would reveal the presence of some unreacted and slightly soluble perchlorate ion from...
the flash powder in this sample, it is unlikely that a statistically significant excess presence of aluminum would be found. To the contrary, by using the morphological criteria for PRRP, the presence of aluminum in the PRRP is unambiguous. This is presented as the third spectrum labeled “3) PRRP”, which is a composite spectrum of 16 pyrotechnic reaction residue particles.

Another potential problem for results from micro-analytical chemistry is that trace quantities of other species might be found that are unrelated to the explosion event, but which might be incorrectly interpreted as originating from the pyrotechnic composition. For example, chlorate ions could originate from weed-killer, and nitrate, sulfate, ammonium and potassium ions could originate from fertilizer.

In this case, only an aluminum peak (from aluminum oxide, Al₂O₃) from the flash powder reaction products is readily seen in the PRRP spectra. Substantial potassium and chlorine peaks (from the potassium chloride [KCl] reaction product) are essentially missing in the composite spectrum, but are weakly present in some of the individual particle spectra. In this case, the virtual absence of the potassium and chlorine peaks is a result of the condensation temperature of potassium chloride being moderate (1478 °C) compared with the high temperature of the fireball and the condensation temperature of aluminum oxide (3528 °C). The result is that the potassium chloride tends preferentially to condense onto the surface of the relatively cool dirt particles that were mobilized as a result of the explosion. As mentioned above, a thorough discussion of this phenomenon is beyond the scope of this paper, but this does help to make the point that knowledge of the expected products of pyrotechnic reactions, and the physical and chemical properties of those products, can be important in correctly interpreting PRRP results.

Note that in this example, neither micro-analytical chemistry nor PRRP analysis alone would be likely to yield completely definitive results about the nature of the pyrotechnic composition involved. Rather it is the combination of information produced by both techniques that can be expected to produce fairly unambiguous results.

Example 2: Use History

An accident was thought to have been caused when a reusable performance appliance was loaded with an inappropriately powerful explosive charge. The appliance was made using an approximately 300-mm (12-inch) length of 102-mm (4-inch) diameter steel tubing having a 6-mm (1/4-inch) wall, welded to a thick steel base plate for stability, as illustrated in Figure 10. This type of appliance can be used to produce both an audible and visual effect by exploding an appropriately sized and constructed pyrotechnic charge within it. Following the incident in question, the appliance was held as evidence and after several months the inside surface of the device was sampled and analyzed for PRRP. The result was that approximately equal numbers of two fairly distinct types of PRRP were found to be present. Their EDS spectra (composites from 8 particles of each type) are shown in Figure 11. One type of PRRP is differentiated by its having an abundance of strontium present with relatively little titanium and aluminum, while the other type had no strontium and an abundance of titanium and aluminum.

![Figure 10. Sketch of the basic construction of a reusable performance appliance used to produce visual and audible effects.](Image)
than what had normally been used. The identification of two distinctively different PRRP supported the theory that there had been a fairly recent change in the type of pyrotechnic composition used in the appliance. Had a single type of pyrotechnic charge been used, one principally producing both titanium rich and strontium-rich residues (in addition to the magnesium and aluminum), then the PRRP would have all been somewhat similar in composition, with the vast majority of PRRP containing substantial and approximately equal quantities of the elements. To the contrary in this case, less than 10% of the PRRP were found to contain approximately comparable quantities of the two sets of characteristic PRRP elements.

Armed with definitive information about the use history of the performance appliance, pyrotechnic crew members acknowledged that both red color producing (strontium containing) devices and much more powerfully explosive titanium flash powder devices had been exploded within the device.

In this case, PRRP analysis produced results that would not have been possible with conventional analytical chemical methods. Had the samples from the appliance been analyzed using conventional micro-analytical chemistry, only a single combined set of results would have been produced.

**Example 3: Incident Chronology**

When an explosion occurs, if the blast or thermal output is sufficient and there are other explosive devices in the immediate area, it is possible for the first explosion to initiate secondary explosions. This occurred in a firework display accident, wherein it was important to identify which of two events occurred first. To simulate such an event on a small scale, a demonstration was staged wherein two relatively small pyrotechnic charges were exploded within 0.2 s of one another. One of the two explosive charges is seen in the upper photograph of Figure 12, and the pair of charges can be seen in the lower photograph at opposite ends of the setup. Each of the charges contained approximately 28 g of composition.

![Figure 11](image1.png)

*Figure 11. Two composite spectra taken from a performance appliance whose misuse was thought to have resulted in an accident.*

![Figure 12](image2.png)

*Figure 12. Photographs showing the setup for a demonstration in which PRRP is used to discover details of the course of an explosion event.*
One charge was a standard fireworks flash powder (70% potassium perchlorate and 30% aluminum powder) while the other was a common theatrical concussion powder, a type of fuel-rich flash powder (50% strontium nitrate and 50% magnesium). The distance between the two charges was approximately 0.5 m (20 inches). The demonstration was conducted in a research bay approximately 4 by 4 by 4 m (12 by 12 by 12 feet) and configured such that one wall was completely removed and open to the outside environment.

After exploding the two charges, the room was cleared of smoke using a large exhaust fan. Once the air had cleared, fragments of the containment vessels (two 35 mm-film canisters, one made from black polyethylene and the other made from uncolored polyethylene) were collected and mounted for PRRP analysis. A total of four specimens were made; each using three small pieces of the same type (color) plastic. Two were made with black plastic, one specimen had the fragments mounted in an orientation such that the inside surface of plastic canister was in an upward orientation, and one specimen with the fragments mounted in an outside-up orientation. (The inside versus outside orientation of the fragments was determined by their direction of curvature.) A similarly-prepared pair of specimens was assembled with the uncolored plastic fragments. In addition a sample was obtained from the midpoint of the light-colored collecting surface, seen in Figure 12, extending between the two steel blocks to which the explosive charges had been attached. (The mostly white strip of collecting material was 3-mm thick tileboard that had been cleaned prior to the test. The pattern of small random spots seen in the upper photo of Figure 12, were permanently imprinted within the surface coating by the tile board’s manufacturer.) This midpoint PRRP specimen was prepared by contact using an adhesive carbon dot as described above.

Based on an initial brief analysis of the PRRP on the sampled items, it was quickly obvious that two chemically different pyrotechnic explosives were involved. Thus, following somewhat the same scheme as in the "Use History" example above, PRRP spectra were divided into different categories. These composite spectra are presented in Figure 13 and are representative of the four categories: “1) Pure Firework Flash”, “2) Mostly Firework Flash”, “3) Mostly Theatrical Concussion”, and “4) Pure Theatrical Concussion”. In this demonstration, 25 PRRP from each of 5 samples were analyzed. Table 1 is a summary of the number of PRRP in each category found on each type specimen.

![Figure 13. A series of four composite spectra representing the four categories of PRRP found in this analysis.](image-url)
Given the geometry of the demonstration setup, it must be expected that there will be a tendency for each exploding charge to project its PRRP outward toward the initial location of the other charge. However, note that the PRRP found on the outside surfaces of both containers were mostly Category 1, having only the components of firework flash powder. Note further that 40% of the PRRP on the outside surface of the black container (that had contained theatrical concussion powder) were also Category 1 (from the firework flash powder). To the contrary, the outside surface of the clear container (that had contained firework flash powder) had no PRRP of Category 4 (pure concussion powder components). This is consistent with the flash powder charge having exploded first, because if the flash powder charge exploded first, its container would no longer be present in the immediate area to collect PRRP from the concussion powder charge when it explodes, even if that second explosion occurred only a small fraction of a second later.

In Table 1, the column labeled “Surface Total” is the total of the PRRP particles collected on the exterior surfaces of the two containment vessels plus those from the midpoint of the collecting surface. Approximately 40% of the PRRP fell into the two “Mixed” categories (Categories 2 and 3). This is an indication of the timing of the two explosions, specifically that there was only a slight delay between the two explosions. (Recall that in this demonstration the two explosions were made to occur within approximately 0.2 s.) From other demonstrations using the same charges and setup, it has been observed that:

- Had the two explosions occurred at the same instant, there would have been more complete mixing of their respective fireballs, and roughly 70% of the PRRP would have contained a significant mixture of the components of both of the pyrotechnic explosives.
- Had the two explosions been more widely separated in time (with as little as 2 s between explosions), there would have been no mixing of the fireballs and only a little mixing in the PRRP. This relatively minor mixing is the result of the remobilization of some of the previously deposited PRRP and would result in roughly 10% of the PRRP containing a mixture of the components of both of the pyrotechnic compositions.

In this example, a thorough analysis of the PRRP reveals that two different types of explosive charges were involved, that two separate explosions took place, that the flash powder charge exploded first, and that the two explosions were only slightly separated in time. There is simply no way in which standard micro-analytical chemical analysis could have produced these results.

**Conclusion**

PRRP analysis will not supplant micro-analytical chemistry as an important analytical tool in the forensic analysis of pyrotechnic incidents. However, at the very least, PRRP analysis provides information that is a useful adjunct to normal micro-analytical chemistry, and there are many occasions when PRRP analysis provides information that is simply beyond the ability of standard micro-analytical chemistry.

**References**


2) K. L. & B. J. Kosanke and R. C. Dujay, “Pyrotechnic Reaction Residue Particle Identifi-

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**Table 1. Number of Each of the Four Types of PRRP on the Items Sampled.**

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<th>PRRP Category</th>
<th>Black Plastic</th>
<th>Clear Plastic</th>
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<td>Outside</td>
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<td>Outside</td>
<td>Inside</td>
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<td>1) Pure Firework Flash</td>
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<tr>
<td>2) Mostly Firework Flash</td>
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<td>5</td>
</tr>
<tr>
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<td>2</td>
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