

## Site exploitation team and mobile lab analyze unknown chemical agents on Thermo Scientific portable instruments



Faced with the challenge of rapidly identifying unknown chemical substances in the field, many civilian, federal and defense agencies use portable spectroscopy to execute their missions. Most often, Raman and Fourier transform infrared (FTIR) spectroscopy are deployed to perform the required analyses.

A tiered sample analysis strategy is common across these agencies. As threatening substances are better defined, greater analytical resources can be brought to bear. While more sophisticated instrumentation may be found in higher levels of agency analysis, portable tools like the Thermo Scientific™ First Defender™ RMX, Thermo Scientific™ TruDefender™ FT, and Thermo Scientific™ Gemini™ analyzers may be appropriate at all levels of analysis. These analyzers are simple to operate in initial field tests, yet sophisticated enough to be beneficial in the laboratory, when used by highly trained operators.

### Profile

The FirstDefender instrument uses Raman technology to identify unknown substances; TruDefender uses FTIR. There are many scenarios across civilian, federal and defense agencies in which Thermo Scientific analyzers are deployed as part of a tiered analysis strategy.

- Military explosive and ordnance disposal units perform on-site analysis. Suspect homemade explosives (HME) and precursor materials are sent to field labs for further analysis, and then escalated to more sophisticated facilities. Each of these operations may use Thermo Scientific portable instruments, based on the sophistication of the user.
- The Federal Bureau of Investigation may perform field testing with Thermo Scientific analyzers before shipping suspect samples to a national laboratory, where the samples are first triaged with the same instruments before being transferred into the lab facility.
- National Guard Weapons of Mass Destruction Civil Support Teams use Thermo Scientific analyzers -- first as a survey team collects samples and again in the Analytical Laboratory Suite, before escalating to other resources, as needed.
- First-responder hazmat teams use Thermo Scientific analyzers for field analysis before escalating to laboratories for environmental or criminal follow up.

Recently, we introduced the Gemini handheld analyzer. Gemini integrates Raman and FTIR technologies into a single, compact instrument. For military and first-responder organizations, the new analyzer is designed to reduce weight and the number of instruments carried downrange while still providing the same level of lab-quality analytical performance.



In 2015, a United States Department of Defense agency compared the Gemini to the FirstDefender and TruDefender analyzers. Following an abbreviated training session, members of a site exploitation team and a mobile laboratory unit assessed the utility and functionality of Gemini – their first use of the instrument – and contrasted its benefits and limitations to the FirstDefender and TruDefender instruments.

### Evaluation Method and Samples

Two operators in Level A personal protective equipment (PPE), and each with a one-hour self-contained breathing apparatus, analyzed unlabeled chemical substances, as presented in various forms and volumes. Operators performed first-tier analysis on eight samples with FirstDefender, TruDefender and Gemini, one sample at a time, for a total of 32 samples. (Level A PPE provides the greatest level of skin, respiratory and eye protection and, thus, substantially impairs senses and movement.)



Later, a second-tier mobile laboratory operator analyzed the same eight samples with the same instruments, under a laboratory hood. In this exercise, the samples were pre-packaged as if a site exploitation team had made the initial analysis and collected evidence.



The eight samples were provided from a simulated, improvised chemical weapons agent production site. With HME and HME precursors, samples included four liquids, two solids, one wet-solid, and one slurry sample. Specifically:

- Thiodiglycol and hydrochloric acid (HCl) liquids for sulfur mustard production.
- Technical grade ammonium nitrate (TGAN) solid, and fuel oil to make ammonium nitrate fuel oil (ANFO).
- Solid urea and liquid nitric acid for urea nitrate (UN).
- On-site production of UN in a paste / slurry form.

During the simulation, our trainers observed sample preparation and

testing protocols. Analytical results were stored on the instruments and subsequently tabulated. Following the exercise, the trainers surveyed the first-time users on their experience with Gemini, taking note of best practices and lessons learned.

### Display of Analytical Results

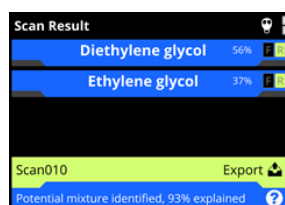
The Thermo Scientific instruments present real-time analytical results by way of colored display screens, which are easy for operators to recognize and read. Colored displays are green, blue, yellow or red, and are identical for each instrument used in the evaluation exercise.

- **Green Screen** – The instrument has matched the tested sample with an item in the instrument library.
- **Blue Screen** – The instrument has identified a mixture of items in the tested sample, with the most prominent item being displayed first.
- **Yellow Screen** – The instrument has identified an item in the sample as “similar” to another item in the instrument library.
- **Red Screen** – The instrument is not able to match the tested sample with any items in the instrument library. In this case, the result screen will prompt the operator to consider using FTIR for analysis.

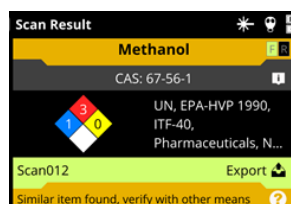
Examples of colored screen displays for the Gemini analyzer are shown below.



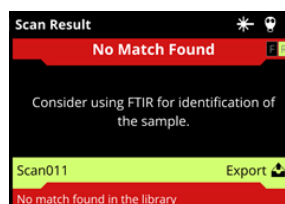
Green Screen – single item match



Blue Screen – mixture match



Yellow Screen – similar item match



Red Screen – no match found

Following the abbreviated training, the new DOD operators in the simulation were presented with eight samples. Each sample was to be scanned with FirstDefender Raman, TruDefender FTIR, Gemini Raman and Gemini FTIR, generating a total of 32 possible tests. Similarly, the second-tier mobile lab operator was presented with the same samples, pre-packaged and submitted as collected evidence by the site team, each to be scanned with the same instruments for another 32 tests.

As the first-time operators analyzed the samples, we observed some deviations from the training. For example, twice operators neglected to run an FTIR background scan. (Prior to each test with an FTIR instrument, a background scan is required.) In another example, an extended period of time elapsed between the background scan, placing the sample on the testing area, and actually performing the analysis. In a Raman test, the operator attempted to scan a small sample on the plastic body of the Gemini instrument, which created “optical noise,” rather than scanning on an appropriate surface, as instructed.



In total, seven tests used procedures outside the scope of the abbreviated training. These were deemed “operator errors,” and were not considered representative of instrument performance. In fairness, our observers recognize that these “errors” occurred following an abbreviated training session. The team believes extended training and experience would serve to minimize these procedural errors and increase operator efficiency.

Additionally, site and lab operators passed on three of the tests, due to known sample size limitations or exercise time. Four of the samples were known to be non-responsive to Raman signal, and the Gemini analyzer correctly indicated that alternate FTIR technology should be applied. A summary of test results is shown in Table 1 (below).

Operator	Correct IDs	Incorrect IDs	Passed Scans	Operator Errors	Adjusted Rate of Correct IDs
Site Team	22	1	5	4	95.7%
Mobile Lab	26	2	2	2	92.9%
Totals	48	3	7	6	94.1%

Table 1

## Feedback from the Operators

Following the staged exercises, our trainers held a debriefing with the operators. Participants offered their opinions openly on the benefits and limitations of the Gemini analyzer, comparing the utility of Gemini to FirstDefender and TruDefender, in some instances.

### Gemini Benefits

- *One unit versus two* – In terms of analytical capacity, data storage and retrieval, and logistics, operators found significant benefit in the integrated Gemini system. A combined Raman and FTIR library, operators said, simplifies analysis downrange and holds the potential to limit Reachback requests. In site exploitation, operators noted that Gemini is lightweight and easier to carry than the FirstDefender and TruDefender combination. And, under the laboratory hood, the Gemini analyzer occupies a smaller footprint, lessening potential contamination due to congested workspace, according to a mobile lab user.
- *Power Supply* – Operators gave positive reviews of Gemini’s tiered, redundant power system. Power is first drawn from a hot-swappable disposable alkaline battery pack. If first-tier batteries are depleted, power is then drawn from an integrated lithium ion battery. If plugged into an 110V outlet, both battery sources are held in reserve.

- *Scan Assist* – The Gemini analyzer guides operators on selection of the appropriate identification technology. Operators said the Scan Assist feature would be valuable, especially in downrange situations, where time-on-site is extended and operators might become fatigued. In addition, they noted, the series of questions posed by Scan Assist could serve as a good reminder for infrequent operators.
- *Profiles* – By setting up Profiles in Gemini, users can customize scan parameters, based on situational awareness and experience – before entering the hazard zone. Operators in the simulation commented that Profiles would be particularly useful if they had received an intelligence report of a suspected agent. If operators have knowledge about the target substance, they could leverage Profiles to ensure compounds of interest are identified and recognized when downrange.
- *Tagging* – Tagging instructs the Gemini instrument, in either Raman or FTIR mode, to look more carefully for certain, specified chemicals. Operators saw general value in Tagging. However, because an “all-hazards” approach is used by many organizations, their ability to use the feature might be limited.
- *Navigation* – Operators reported that Gemini operation is easy to follow. Even when wearing Level A PPE, the instrument’s touch screen worked well in the scenarios and operators noted simple, functional navigation.
- *Probe* – Compared to the FirstDefender RMX, the fixed but flexible probe for Gemini Raman analysis is shorter, smaller and more flexible. Operators noted that this improves ease of use and maneuverability. Gemini also has a reduced eye-safety distance of 13 inches, compared to 17.25 inches for RMX, allowing the operator to be closer to the work.

### Gemini Limitations

- *Multiple Options* – The Gemini instrument comes with multiple features – two spectroscopy technologies, Profiles, FTIR Scan Delay, and motorized anvil, to name a few. These options, in some situations, may lead to confusion, operators said. However, with training provided by us – and practice in field simulations – operators can learn to leverage these features for their advantage.
- *Vial Compartment* – The original Thermo Scientific™ Ahura FD™ (not used in the simulation) had a vial compartment that tilted the vial 45 degrees. This caused small-sized samples of one or two drops to collect in the “corner” of the vial, directly in the path of the Raman laser. Operators noted that the Gemini and RMX vial holder is upright, not angled, and that the path of the laser is above the sample. However, tilting the Gemini or RMX instrument to 45 degrees will produce a successful scan on a two-drop sample.
- *Pre-crushing with FTIR* – Site and lab operators observed that samples presented for FTIR analysis need to be pre-crushed in order to obtain accurate results, and that the motorized anvil, alone, cannot do all the sample preparation. While it is correct that the anvil crushes a sample against the diamond ATR (part of the spectroscopy system), the instrument can only operate as well as the sample that is presented. A lump sample with a coating, for example, will produce a lower quality result than the same sample that is “pre-crushed,” allowing the instrument to get a good look at the center and more representative part of the lump, not just the coating. *Note: Since the simulation occurred, we have redesigned the profile of the anvil tip. The redesign has shown significant*

performance improvement when scanning such hard crystalline materials as ammonium nitrate prill, crystalline, and potassium chlorate.

## Best Practices and Lessons Learned

A general discussion between Thermo Fisher trainers and the operators highlighted several best practices in the process of using Thermo Scientific analyzers to identify unknown chemical substances. These included:

- Scotch™ Brand transparent tape with a matte finish proved effective for sampling very small (pinhead size) solid samples with both Raman and FTIR technology. When using the Raman laser in point-and-shoot mode, the tape can be used to suspend the sample in air and eliminate background interference. The same sample can then be positioned exactly on the FTIR diamond surface and held by the tape before applying anvil pressure for a second test.



- Pre-crushing hard, solid samples often improves results with FTIR. This assures better contact against the diamond surface. (The new Gemini anvil tip also improves results.)
- Altering FTIR pressure may affect results for solid / solid and solid / liquid mixtures. Shape and hardness of solid particles will affect contact on the diamond at various pressures. Liquids can be forced out of solids with high pressure, thus altering the proportion of solid ingredient to liquid ingredient, and altering spectroscopic signal return.
- Operators should consider physical separation of mixtures to test substances separately with Raman and FTIR. Physically separating mixtures presents two or more simple tests for the instruments rather than one complex mixture.

## Conclusion

Many first-responder, federal and defense agencies use portable Raman and FTIR spectroscopy to identify unknown chemical substances. The Thermo Scientific FirstDefender, TruDefender, and now, Gemini analyzers are often deployed at multiple levels of a tiered substance analysis system within these agencies.

In a simulation with our trainers, following an abbreviated training session, a United States DOD agency tested unknown chemical substances with all three Thermo Scientific instruments, noting the benefits and limitations of the new Gemini. Operators in the simulation included a site exploitation team and a mobile laboratory.

Adjusting for passes and “operator errors” across 64 possible samples, the Thermo Scientific instruments correctly identified 94.1 percent of the samples presented. Importantly, in the opinion of the observers, extended training and experience would serve to minimize procedural errors and increase operator efficiency.

While all three Thermo Scientific instruments performed equally in the analysis of samples, operators reported that Gemini offered significant user benefits. These included such new features as the integration of Raman and FTIR into a single device, an extended and redundant power supply, Scan Assist and Profiles, a modified probe, and enhanced navigation. Operators also noted Gemini limitations. These included the possibility that Gemini’s multiple features might confuse an operator, an upright vial compartment requires tilting the analyzer to scan tiny samples, and the potential need to pre-crush solids for FTIR (though a new Gemini anvil design has improved performance with this generally accepted FTIR limitation).

*The DOD agency participated in this study for the purpose of objectively gathering user information and collecting data. There is specifically no endorsement of Thermo Scientific instruments of any kind.*

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