

# PROCEEDINGS OF SPIE

[SPIDigitalLibrary.org/conference-proceedings-of-spie](https://spiedigitallibrary.org/conference-proceedings-of-spie)

## High-speed and large-area scanning of surfaces for trace chemicals using wavelength-tunable quantum cascade lasers

David B. Kelley, Derek Wood, Anish K. Goyal, Petros Kotidis

David B. Kelley, Derek Wood, Anish K. Goyal, Petros Kotidis, "High-speed and large-area scanning of surfaces for trace chemicals using wavelength-tunable quantum cascade lasers," Proc. SPIE 10629, Chemical, Biological, Radiological, Nuclear, and Explosives (CBRNE) Sensing XIX, 1062909 (16 May 2018); doi: 10.1117/12.2304387

**SPIE.**

Event: SPIE Defense + Security, 2018, Orlando, Florida, United States

# High-speed and large-area scanning of surfaces for trace chemicals using wavelength-tunable quantum cascade lasers<sup>1</sup>

David B. Kelley, Derek Wood, Anish K. Goyal<sup>2</sup>, and Petros Kotidis

Block MEMS, 132 Turnpike Road, Southborough, MA 01772

## ABSTRACT

A standoff chemical detection system is being developed to detect and identify a wide range of trace chemicals on a variety of natural and artificial surfaces. The system is based on active mid-infrared (MIR) hyperspectral imaging in which the target surface is illuminated using miniature, rapidly tunable, external-cavity quantum cascade lasers (EC-QCLs). These lasers are tuned across the wavelength range of 7.7 – 11.8  $\mu\text{m}$  while a HgCdTe camera captures images of the reflected light. Hypercubes with 128x128 pixels and more than 130 wavelengths are captured within 0.1 s. By operating the camera in sub-window mode, hypercubes with 16x96 pixels and 138 frames are captured in only 14 ms. To the best of our knowledge, these represent the world's fastest acquisition of active MIR hypercubes. Raster-scanning of the laser beam is used to scan large regions. In this talk, we will present results for detecting traces of solid chemicals (with loadings on the order of 100  $\mu\text{g}$ ) on natural outdoor surfaces such as roofing shingles, concrete, sand, and asphalt at a standoff distance of 5 m. The measured spectra are found to correlate very well with those of reference measurements made of pure chemicals after accounting for the substrate reflectance.

**Keywords:** Mid-IR spectroscopy, Quantum cascade laser, Hyperspectral imaging, Chemical detection, HgCdTe focal plane array

## 1. INTRODUCTION

Mid-infrared (MIR) spectroscopy is one of the most promising methods for the standoff detection of trace surface chemical due to the fact that most substances have strong and unique absorption features in the MIR portion of the optical spectrum [1-7]. Our detection approach, called active MIR hyperspectral imaging (HSI), involves the use of wavelength-tunable lasers in combination with a high-speed camera to capture hyperspectral images (i.e., hypercubes) of the reflectance of the target surface.

As shown in Figure 1, a wavelength-tunable MIR laser illuminates the target of interest and the reflected light is captured by a camera. As the laser wavelength is tuned, the camera synchronously captures images of the reflected light. The raw hypercube is processed to correct for the background thermal radiation and the intensity pattern of the illuminating laser beam to generate a hypercube that represents the reflectance of the target surface. The reflectance hypercube is then analyzed and compared to a reference library of spectral signatures to generate a detection map which identifies and spatial maps any chemical contamination on the surface of the target. As shown in the figure, it is also possible to detect the presence of gases that may be present in the beam path.

---

<sup>1</sup> This research was funded by the Office of the Director of National Intelligence (ODNI), Intelligence Advanced Research Projects Activity (IARPA), through the AFRL contract FA8650-16-C-9107. All statements of fact, opinion or conclusions contained herein are those of the authors and should not be construed as representing the official views or policies of IARPA, the ODNI, or the U.S. Government. The Government is authorized to reproduce and distribute reprints for Governmental purposes notwithstanding any copyright annotation thereon.

<sup>2</sup> Corresponding author: anish.goyal@blockeng.com.

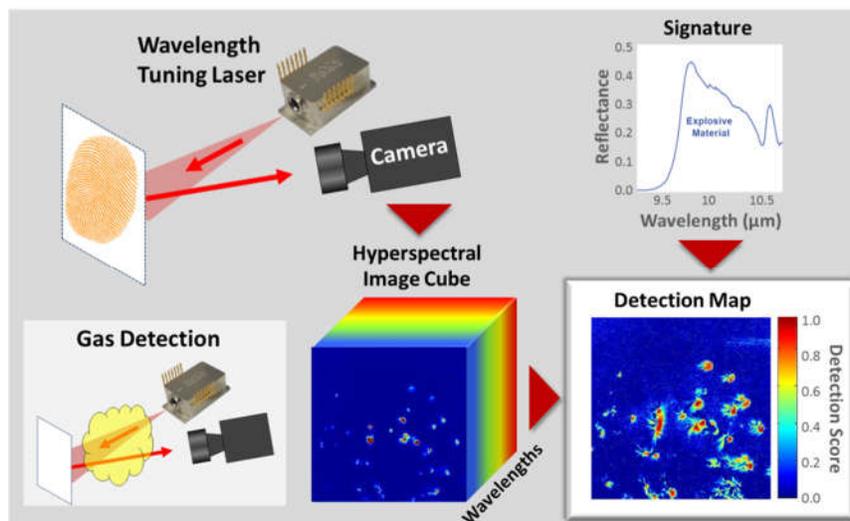


Figure 1. Concept of hyperspectral imaging using a wavelength-tunable MIR laser.

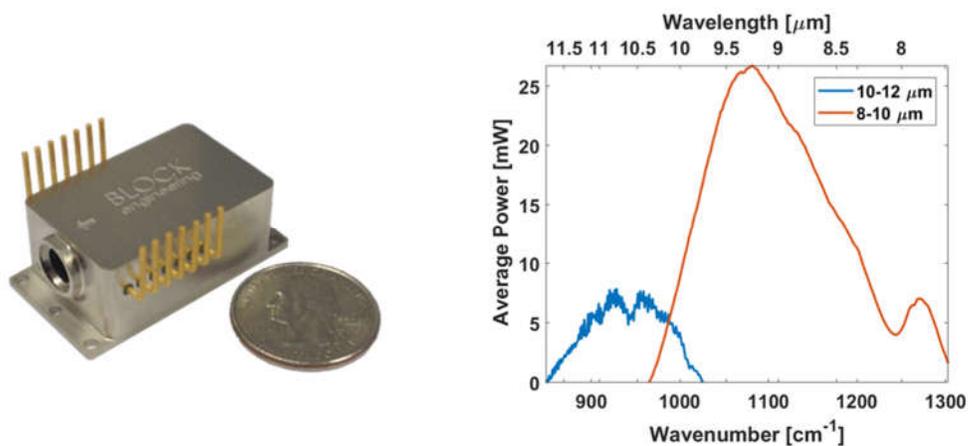


Figure 2. *Left:* Block's Mini-QCL™ is a small, rapidly tunable external-cavity QCL. *Right:* Power spectrum of the two QCLs used in this work as measured at 5% duty cycle with 50-ns pulses.

## 2. HYPERCUBE ACQUISITION

External-cavity quantum cascade lasers (EC-QCLs) are used for illumination of the target [8]. These are based on Block Engineering's Mini-QCL™ shown in Figure 2 which is a miniature, widely tunable, high-speed, and rugged EC-QCL. They are commercially available in wavelengths from about 5.4 to 13  $\mu\text{m}$ . Our system currently uses two Mini-QCLs whose output is combined using beam splitters. Figure 2 plots the average power versus wavelength for the two lasers at a duty cycle of 5%. The lasers are tunable across wavelength ranges of 7.7 – 10.3  $\mu\text{m}$  ( $1300 - 970 \text{ cm}^{-1}$ ) and 9.8 – 11.8  $\mu\text{m}$  ( $1020 - 850 \text{ cm}^{-1}$ ) and provide average powers of 26 mW and 7 mW, respectively. Future system enhancements will include multiplexing additional lasers to increase the spectral range and incorporating lasers that can generate about 50 times more power.

For image capture, the system uses a LN<sub>2</sub>-cooled HgCdTe focal-plane array (FPA) with 40  $\mu\text{m}$  pixels which is capable of capturing 128x128 pixel frames at up to 1600 frames per second (fps). In sub-window mode, frames with 16x96 pixels can be captured at 9,400 fps.

Both laser and camera triggering are controlled by a fast digital-input/output (DIO) module such that frame capture is synchronized with laser illumination for each wavelength. The results of a typical measurement sequence are shown in

Figure 3. This particular measurement uses only one laser, but the process is similar for the case where multiple lasers are used. The EC-QCL is swept in wavelength while the camera captures a total of 150 frames within 0.1 s. Of those frames, the laser is illuminated the surface with different wavelengths for 137 frames while 13 frames are 'dark' without laser illumination. The dark frames are used to measure, and then subtract, the passive thermal background. For this measurement, the sample consists of roughened aluminum which is partially contaminated with silicone grease. The optics are arranged to image the sample with 128 x 128 pixels with a high spatial resolution of 80  $\mu\text{m}$ . The measured area of 1  $\text{cm}^2$  is fully illuminated by the laser beam. The reflection spectra from the clean and contaminated areas that are derived from the reflectance hypercube clearly indicate the absorption peaks associated with silicone at 1025, 1075, and 1260  $\text{cm}^{-1}$ .

For measurement of larger surfaces, the system has an integrated two-axis galvanometer-based scanning mirror system (scanner) to raster scan the laser illumination across the surface. This is graphically depicted in Figure 4. Raster scanning also allows one to choose a smaller beam size (and thus higher average fluence) at the expense of a longer total capture time.

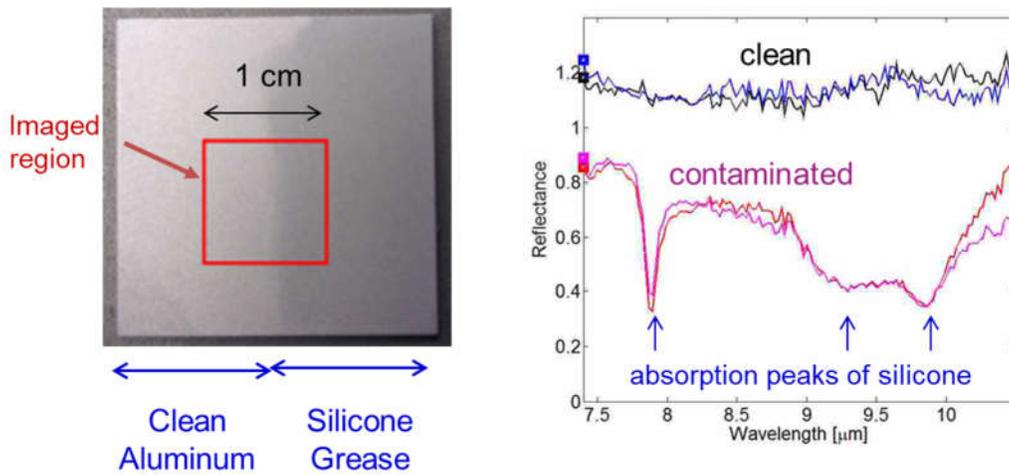


Figure 3. *Left*: Photograph of roughened aluminum sample that is partially contaminated with silicone grease. *Right*: Reflectance spectrum of the clean and contaminated areas as derived from a measured reflectance hypercube.

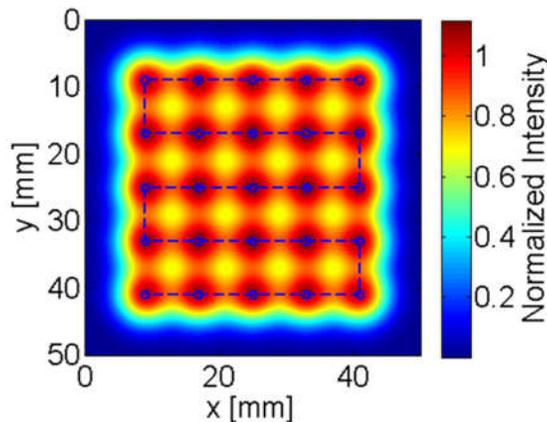


Figure 4. Graphical simulation of raster-scanning the laser beam to measure a larger surface area.

### 3. RESULTS

Measurements have been made of a large number of samples using the HSI system. A few examples are given here.

#### 3.1 Solid traces on outdoor surfaces

Traces of solid powders were applied to a variety of outdoor surfaces using a dry-transfer technique. The surfaces include stone roofing shingles, concrete, asphalt, and sand. Figure 5 shows measurement results for 100  $\mu\text{g}$  of caffeine applied to a roofing shingle and measured at a standoff distance of 0.4 m. For these measurements, two EC-QCLs were used to acquire hypercubes that span the wavenumber range of 850 – 1300  $\text{cm}^{-1}$ .

It was determined that the reflectance of the contaminated portions of the sample fit very well the library spectrum for caffeine powder after normalizing the measured reflectance by the reflectance of the underlying substrate. As shown in Figure 5, after applying this normalization, a detection score was then associated with each pixel in the hypercube based on its spectral similarity with the reflectance of caffeine powder and a threshold was then applied to create a detection map.

Figure 6 shows detection results for traces of caffeine on various surfaces as measured at a standoff distance of 5 m. Figure 7 demonstrates the ability of the system to distinguish between and identify three different chemicals on concrete at a standoff distance of 5 m.

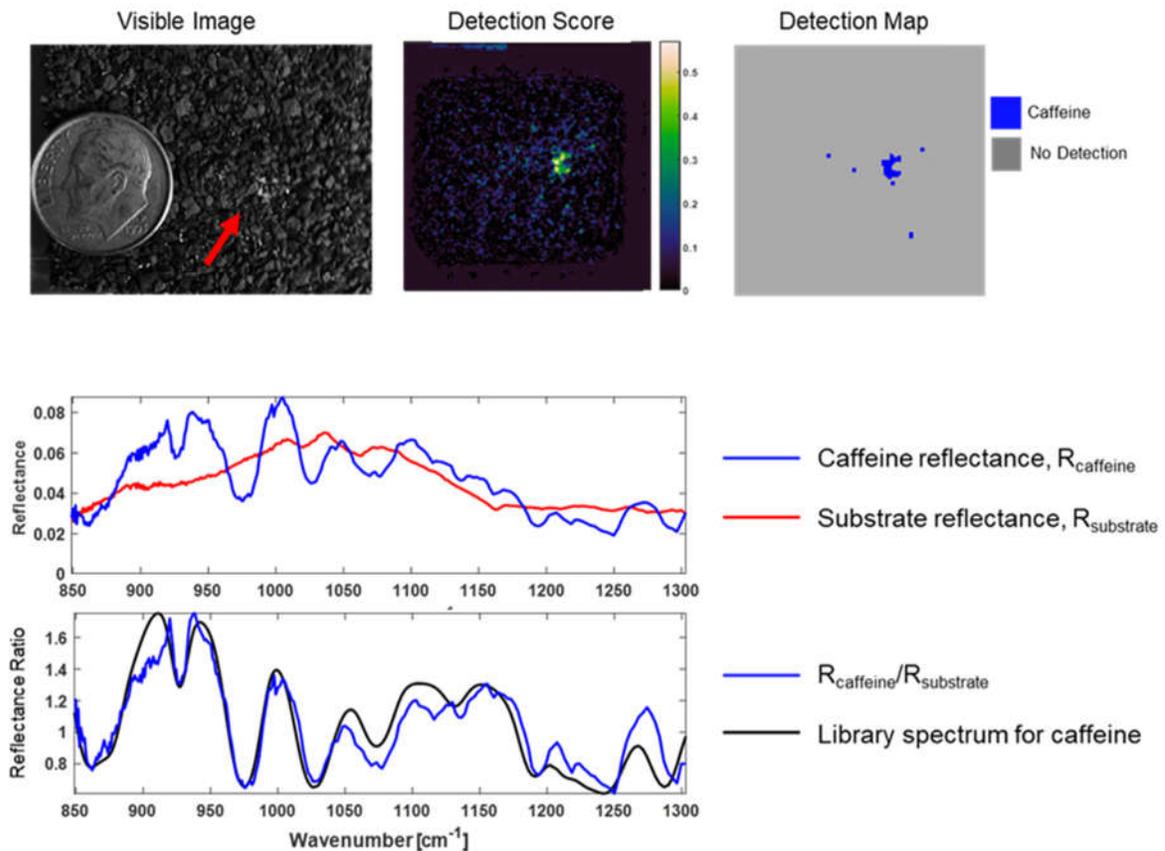


Figure 5. Measurement results for detection of 100  $\mu\text{g}$  of caffeine powder on roofing shingles at a standoff distance of 0.4 m.

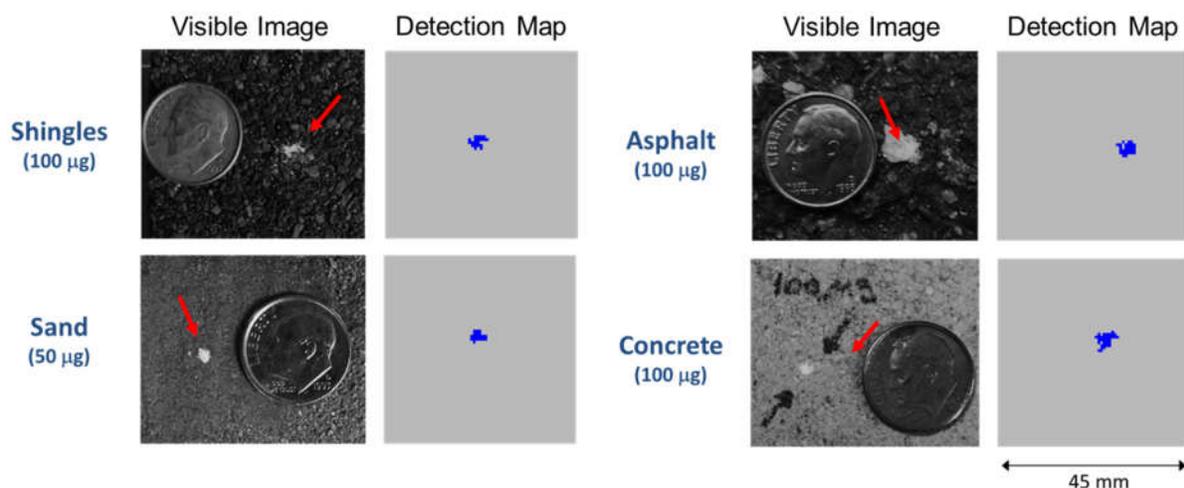


Figure 6. Detection of caffeine powder traces (between 50 – 100  $\mu\text{g}$ ) on various surfaces at a standoff distance of 5 m.

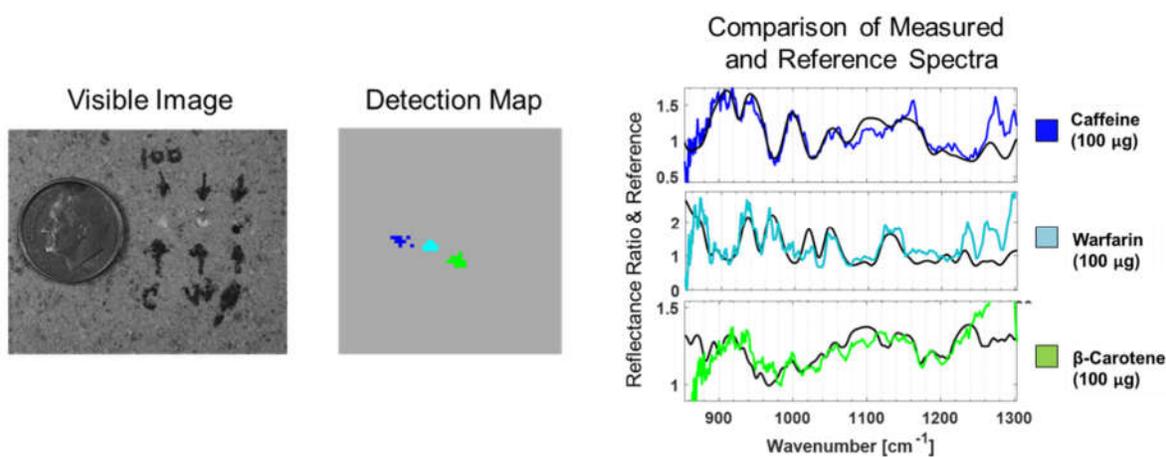


Figure 7. Detection and identification of 3 different chemicals applied to concrete at standoff distance of 5 m.

### 3.2 Very high-speed acquisition

In order to demonstrate the very high-speed acquisition of hypercubes, a sample was prepared in which each letter of the name “BLOCK” was created in a different chemical on a glass substrate as shown in Figure 8. The chemical concentration was uncontrolled and relatively high. A composite hypercube of this sample was obtained as follows. The laser beam was raster scanned across the target in 5 steps (one for each letter). The size of the composite hypercube was  $16 \times 96$  pixels such that the camera could be operated at a frame-rate of 9,400 fps. A hypercube with a total of 138 frames (105 of which contain unique illumination wavelengths) was acquired at each spot within 14 ms. To the best of our knowledge, this represents the world’s fastest acquisition of an active MIR hypercube. The total time required to raster the laser beam across the five points and acquire the raw hypercube was only 74 ms. The measured reflection spectra for the various chemicals matches the expected reflectance except for deviations in the wavenumber range  $850 - 900 \text{ cm}^{-1}$ . We are currently working to understand the cause of this deviation. Nevertheless, we believe that this demonstration illustrates the ability of MIR HSI to very rapidly scan surfaces for trace chemicals.

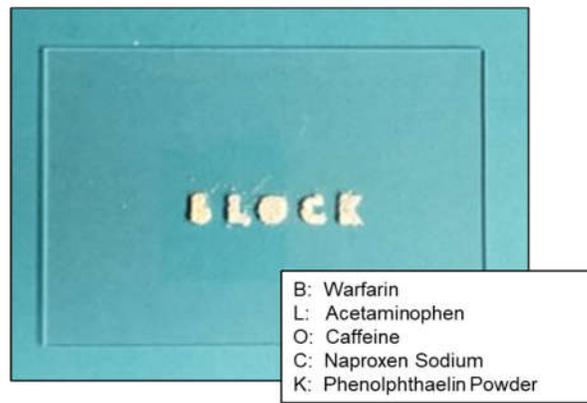


Figure 8. Sample in which each letter of the name “BLOCK” is written in a different chemical on a glass substrate.

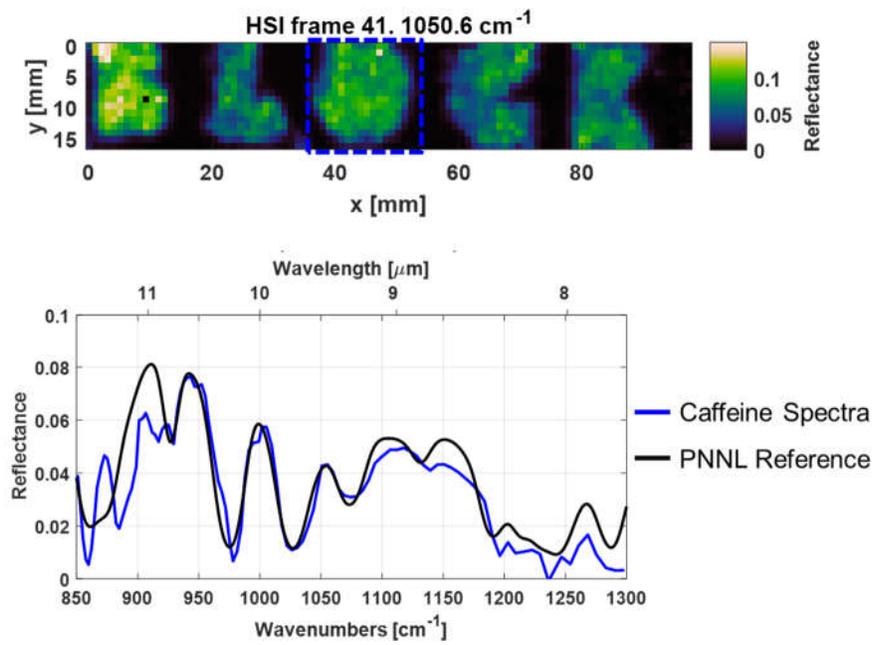


Figure 9. *Top*: Reflectance of the sample at  $1050.6 \text{ cm}^{-1}$ . *Bottom*: comparison of the measured and reference spectra for caffeine.

## 4. CONCLUSIONS

We present results from a hyperspectral imaging system that accurately and rapidly captures hypercubes using EC-QCLs. The detection and identification of solid traces on various outdoor surfaces is demonstrated at a standoff distance of up to 5 m. Also, the speed of the detection system has been increased so that hypercubes can be acquired at the maximum frame-rate of the camera of 9,400 fps. A composite hypercube with 16 x 96 pixels that was obtained by sequential illumination 5 spots on the sample surface was acquired in only 74 ms. We believe that this represents the highest speed acquisition of laser-based MIR hypercubes to date.

## 5. ACKNOWLEDGEMENTS

This work has been supported by the IARPA SILMARILS program through AFRL contract FA8650-16-C-9107. We appreciate the support and expert guidance of the SILMARILS program manager, Dr. Kristin DeWitt. We also thank the Applied Physics Laboratory at Johns Hopkins University (JHU/APL), Naval Research Laboratory (NRL), and the Pacific Northwest National Laboratory (PNNL) for providing test samples and reference spectral data. We also acknowledge numerous interactions with our SILMARILS team members from Alpes Lasers (Richard Maulini) and Systems & Technology Research (Gil Raz, Cara Murphy, Mark Chilenski, and Robert Argo).

## REFERENCES

- [1] A. K. Goyal and T. R. Myers, "Active mid-infrared reflectometry and hyperspectral imaging", chapter in [Laser-Based Optical Detection of Explosives] CRC Press (2015).
- [2] Goyal, A. K., et al., "Active infrared multispectral imaging of chemicals on surfaces," Proc. SPIE 8018, 80180N (2011).
- [3] Goyal, A. K., et al., "Active hyperspectral imaging using a quantum cascade laser (QCL) array and digital-pixel focal plane array (DFPA) camera," Opt. Express 22, 14392 (2014).
- [4] Ostendorf, R., et al., "Recent Advances and Applications of External Cavity-QCLs towards Hyperspectral Imaging for Standoff Detection and Real-Time Spectroscopic Sensing of Chemicals," Photonics, 3(2), 28 (2016).
- [5] Kelley, D. B., et al., "High-speed mid-infrared hyperspectral imaging using quantum cascade lasers," Proc. SPIE 10183, 1018304 (2017).
- [6] Myers, T., et al., "Mid-infrared hyperspectral simulator for laser-based detection of trace chemicals on surfaces," Proc. SPIE 10198, 101980C (2017).
- [7] Raz, G., et al., "Novel trace chemical detection algorithms: A comparative study," Proc. SPIE 10198, 101980D (2017).
- [8] Faist, J. [Quantum Cascade Lasers], Oxford University Press: Oxford, UK, (2013).