Forensic Technology Testing & Evaluation Form

Forensic Technologies Center of Excellence (FTCoE)

Cooperative Agreement Award #2008-MU-MU-K003

Final Report Date: 02/10/2010

Forensic Technology Testing & Evaluation Project

Project Title:	**Projected Start Date:**
RxSpec® 700Z Spectrometer Evaluation	5/13/09
Evaluation Type:	**Projected End Date:**
Portable Instrument (for chemical identification)	11/9/09
Evaluation Team Leader:	**Contact Telephone:**
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Evaluation Team:

Name	Title	Email	Phone
See contact info above			

Manufacturer Information for product(s) being evaluated:

Manufacturer	Address	Contact Person	Phone
ASD Inc.	2555 55th Street, Suite 100 Boulder, CO 80301 USA	Dan Shiley	(303) 444-6522

Evaluation Overview

Evaluation Summary:
The Forensic Services – Chemistry section of the National Forensic Science Technology Center (NFSTC) performed a brief assessment on the RxSpec® 700Z spectrometer manufactured by ASD, Inc. This type of visible/near infrared (Vis/NIR) spectrometer is currently being utilized to conduct fast, non-destructive, qualitative or quantitative analysis on solids, powders and other materials in a variety of different industries, including pharmaceutical manufacturing, polymer recycling, agriculture, environmental monitoring, and petrochemical production. Given that Vis/NIR has been well established and scientifically proven for use in material identification for these diverse industries, this brief performance evaluation was conducted to determine whether the RxSpec 700Z spectrometer is currently suitable for the analysis and identification of controlled substances while being utilized in traditional and non-traditional laboratory-based environments. The RxSpec 700Z is a field-portable Vis/NIR spectrometer that is housed in a durable protective aluminum case that allows for easy instrument relocation and setup. The unit is capable of utilizing a 110–120V AC or a 12V DC internal or external power source. The instrument uses a halogen lamp as the excitation incident light source which is focused onto the unknown sample from underneath the sampling window. When a sample is exposed to the electromagnetic radiation emitted from the light source, that radiation will be reflected, absorbed and/or transmitted. The interaction between this radiation and the compound(s) contained within the sample cause molecular excitation as well as vibrational energy changes. These vibrational changes cause functional groups, especially O-H, C-H, C-O, C=O, C=C, S-H and N-H if they are present within the sample compound(s), to stretch and bend. During the analysis, the instrument collects digitized data of the corresponding overtones and combinations of vibrations that result, which in turn provides a spectra or unique chemical “fingerprint” of the material. This chemical “fingerprint” is represented in a graphical plot of the spectral reflectance versus the wavelength. The RxSpec 700Z is capable of scanning a wavelength range from 350 nm to 2500 nm with a spectral resolution of 3 nm in the visible region and 10 nm in the NIR. In order to keep this evaluation project manageable and to finish it within the given time frame, only controlled substances, cutting agents and mixtures thereof were utilized in this assessment. The objectives for this evaluation were as follows: 1. Build a chemometrics model for controlled substances identification using spectra obtained from samples of controlled substances, cutting agents, and non-controlled substances at varying ratios ranging from 100:0 to 5:95. 2. Objectively assess and determine the relevance of the RxSpec 700Z spectrometer for the analysis and identification of controlled and non-controlled substances. 3. Perform experiments to determine specificity, sensitivity, reproducibility, portability and the effects of environmental factors on the instrument’s performance. 4. Provide user feedback to the forensic community as well as to the manufacturer regarding assessment findings.

Experimental Design:

To properly assess the application of the RxSpec 700Z spectrometer for use in controlled substance identification, experiments were specifically designed to thoroughly and objectively test the analyzer's performance. To accomplish this, the assessment was divided into the following phases:

Phase I: Development of a Chemometrics Model for Classification/Identification of Controlled Substances

- Reference samples consisting of mixtures of controlled substances and selected cutting agents were made ranging from ratios of 5:95 through 100:0 (weight to weight).
- A U.S. Pharmacopeia (USP) standards test was performed daily on the spectrometer to check wavelength accuracy, photometric linearity, and photometric noise.
- Reference samples were placed into appropriately labeled 20-mL disposable scintillation vials and capped.
- The reference samples of pure controlled substances, pure cutting agents and mixtures thereof were analyzed as reference spectra (refer to Table 1 on the following page).
- A baseline was taken between samplings using the white reference standard provided with the instrument.
- A minimum of twenty spectra were collected for each reference standard.
- The PLSplus/IQ Navigator application within a popular spectroscopy software suite of GRAMS/AI v8.0 software was used to construct a chemometrics model using the spectra obtained from reference standards. This mathematical model significantly reduces any molecular variability occurring within the reference spectra, thus limiting the occurrence of false positive and false negative results.

Phase II: Determination of Sensitivity, Specificity, Reproducibility, Portability, and Environmental Effects

- Sensitivity: Not performed.*
- Specificity: Specificity will be assessed using cutting agents, non-controlled substances, training samples and other controlled substances not used in the construction of the chemometrics model (refer to Table 3 on the following page).*
- Intra-day/ten-day inter-day reproducibility study: Not performed.*
- Portability: Not performed.*
- Environmental effects: Not performed.*

  **Experiments were either discontinued or not performed due to the high rate of misidentification obtained using the developed chemometrics model(s).*

Phase III: Testing of the RxSpec 700Z and the Developed Chemometrics Model on Real Case Samples

- The instrument was transported to the Manatee County Sheriff's Office and set up, and a USP test was performed on the instrument.
- Adjudicated forensic drug case samples were transferred to appropriately labeled 20-mL scintillation vials and capped.
- The following information was recorded for each case sample before analysis was conducted with the RxSpec 700Z:
  - Forensic laboratory identification number
  - Methodology utilized for laboratory identification and confirmation
  - Reported forensic laboratory identification
- Each sample was analyzed on the RxSpec 700Z in triplicate, and test results were recorded.

Standards, Controls and Samples Interrogated During Evaluation:

Table 1: Reference Standards for Chemometrics Model Build

Controlled Substance	Cutting Agent	Ratio(s)
Heroin	Caffeine	5:95, 20:80, 40:60, 50:50, 80:20
	Procaine	
	Quinine	
Methamphetamine	Caffeine	
	DMS	
	Niacinamide	
	Benzocaine	
Cocaine Base	Caffeine	
	Mannitol	
	Boric acid	
Cocaine HCl	Caffeine	
	Mannitol	
	Procaine	
Heroin	None	100:0
Methamphetamine		
Cocaine Base		
Cocaine HCl		
None	Niacinamide	0:100
	Boric Acid	
	Benzocaine	
	Caffeine	
	Procaine	
	Inositol	
	Tetracaine	
	Lidocaine	

Table 2: Samples for Sensitivity Determination

Controlled Substance	Cutting Agent	Ratio(s)
Heroin	Caffeine	80:20, 70:30, 60:40, 50:50, 40:60, 30:70, 20:80, 10:90, and 5:95
Methamphetamine		
Cocaine Base		
Cocaine HCl		

Table 3: Specificity Samples

Training Samples (Cases)	Controlled Substances
d,l-Amphetamine, TS-001	Methylenedioxyamphetamine (MDA)
Cocaine HCl, TS-002	Methylenedioxyethylamphetamine (MDEA)
Cocaine base, TS-003	Morphine
MDMA/Methamphetamine, TS-006	Codeine Sulfate

Cutting Agents	Non-Controlled Substances
Caffeine	Acetylsalicylic Acid
Procaine	Ibuprofen
Mannitol	Guaifenesin
Boric Acid	Diphenhydramine
	Chlorpheniramine
	Pseudoephedrine

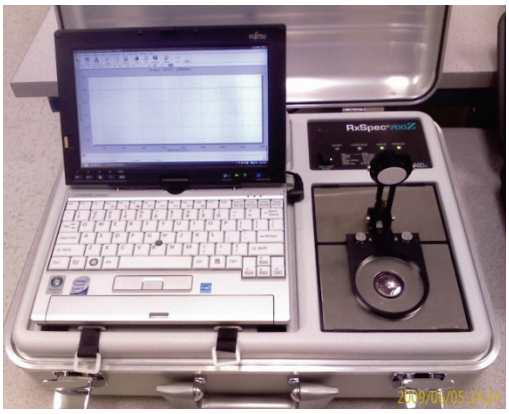
Product(s) Specifications:

Product Name(s)	Model Number:	Serial/Lot Number:	Dimensions:
RxSpec 700Z Spectrometer	700Z 350-2500	11005	6in x 13in x 18in
	Cost:	**Weight:**	**Power Req.:**
	Approximately $55,000	**22 lbs**	**AC Input 100–240V, DC 12V, or 12V NiMH battery**
Storage Conditions	**0 to 45°C**		
Operational Conditions:	**5 to 35°C**		
Associated costs: *(consumables, maintenance, etc.)*	**Disposable scintillation sample vials – $203/case of 500.**		

Brief Description of Product(s)/Technology/Procedure Being Evaluated:

The RxSpec 700Z spectrometer manufactured by ASD, Inc., is a field-portable Vis/NIR spectrometer that is housed in a durable protective aluminum case that allows for easy instrument relocation and setup. The unit is capable of utilizing a 110–120V AC or a 12V DC internal or external power source. The instrument uses a halogen lamp as the excitation incident light source; the light is focused onto the unknown sample from underneath the sampling window. During the analysis, the instrument collects digitized data on the vibrations that result from the molecular excitation of the sample, which in turn provides a spectra or unique chemical “fingerprint” of the material.

Photo/Image of Product(s):



Evaluation

Instrument Setup Performed by:

_____ Manufacturer
__X__ Manufacturer and Evaluator(s)
_____ Evaluator(s) Only

Instrument Setup Comments:

The instrument arrived at the NFSTC laboratory housed in a secondary Pelican™-type case. The unit was removed from the transport case and set up. Power was provided to the RxSpec 700Z instrument using a plug and a 110–120V AC outlet. The instrument was powered on and allowed to warm up for 10 minutes before a USP test was performed using the set of seven different Gray Scale Standards (pucks) from Avian Technologies.

Level of Operator Knowledge as Set by Manufacturer:

_____ Non-Scientist
__X__ Technician
_____ Scientist

Results of Evaluation (Tables, Graphs)

With assistance from a representative of the manufacturer, the evaluator developed a chemometrics model using GRAMS/AI software and spectra obtained from analyzing known standard samples. Below are some of the results obtained from samples analyzed during the Specificity portion of the evaluation.

Material Report

Date: 8/7/2009 12:09:36 PM Operator: Kirk M. Grates
Instrument:<unknown> Serial No.: 11005
Sample: Aspirin
Comments:

Constituent	Concentration	ID	M-Distance
Cocaine Base discriminant		Fail	288.52
Cocaine HCL		Fail	137.263
DMS		Fail	4.17E+05
Heroin		Fail	689.022
Lidocaine_SG1 31_MSC_750-2450		Fail	1.46E+06
Mannitol_SG1 31_MSC_750-2450_1		Fail	2.97E+05
Meth	116.442 1		385.976
Niacinamide_SG1 31_MSC_750-245		Fail	3.45E+05
Procaine		Fail	1.29E+05
Tetracaine		Fail	2.70E+04
Benzocaine		Fail	5.96E+05
Boric_Acid		Fail	6.91E+05
Caffeine_SG1 31_MSC_550-2450		Fail	4.27E+04

Material Report

Date: 8/7/2009 12:12:12 PM Operator: Kirk M. Grates
Instrument:<unknown> Serial No.: 11005
Sample: Benzocaine
Comments:

Constituent	Concentration	ID	M-Distance
Cocaine Base discriminant		Fail	21.759
Cocaine HCL		Fail	263.52
DMS		Fail	1.69E+07
Heroin		Fail	346.31
Lidocaine_SG1 31_MSC_750-2450		Fail	1.87E+06
Mannitol_SG1 31_MSC_750-2450_1		Fail	3.15E+06
Meth	-150.726 1		1.26E+03

Niacinamide_SG1 31_MSC_750-245	Fail	4.47E+04
Procaine	Fail	1.11E+05
Tetracaine	Fail	3.46E+03
Benzocaine	Fail	157.082
Boric_Acid	Fail	6.14E+05
Caffeine_SG1 31_MSC_550-2450	Fail	5.53E+05

Material Report

Date: 8/7/2009 11:54:35 AM Operator: Kirk M. Grates
Instrument:<unknown> Serial No.: 11005
Sample: Boric Acid
Comments:

Constituent	Concentration	ID	M-Distance
Cocaine HCL		Possible	1.275
Cocaine Base discriminant		Fail	178.19
DMS		Fail	2.39E+07
Heroin		Fail	1.73E+04
Lidocaine_SG1 31_MSC_750-2450		Fail	1.25E+07
Mannitol_SG1 31_MSC_750-2450_1		Fail	1.25E+04
Meth	136.102 1		682.225
Niacinamide_SG1 31_MSC_750-245		Fail	2.82E+05
Procaine		Fail	2.57E+06
Tetracaine		Fail	8.31E+06
Benzocaine		Fail	1.80E+06
Boric_Acid		Fail	57.088
Caffeine_SG1 31_MSC_550-2450		Fail	3.69E+05

Material Report

Date: 8/7/2009 12:06:17 PM Operator: Kirk M. Grates
Instrument:<unknown> Serial No.: 11005
Sample: Caffeine
Comments:

Constituent	Concentration	ID	M-Distance
Cocaine Base discriminant		Pass	1.553
Cocaine HCL		Fail	3.692
DMS		Fail	2.80E+05
Heroin		Fail	3.799
Lidocaine_SG1 31_MSC_750-2450		Fail	1.66E+06
Mannitol_SG1 31_MSC_750-2450_1		Fail	1.25E+05

Meth	-3.614 1		3.076
Niacinamide_SG1 31_MSC_750-245		Fail	5.49E+05
Procaine		Fail	1.22E+05
Tetracaine		Fail	2.63E+04
Benzocaine		Fail	3.97E+06
Boric_Acid		Fail	2.91E+05
Caffeine_SG1 31_MSC_550-2450		Fail	29.138

Material Report

Date: 8/7/2009 11:59:16 AM Operator: Kirk M. Grates
Instrument:<unknown> Serial No.: 11005
Sample: Mannitol
Comments:

Constituent	Concentration	ID	M-Distance
Cocaine HCL		Possible	2.622
Cocaine Base discriminant		Fail	15.21
DMS		Fail	3.90E+08
Heroin		Fail	2.80E+03
Lidocaine_SG1 31_MSC_750-2450		Fail	9.79E+05
Mannitol_SG1 31_MSC_750-2450_1		Fail	10.951
Meth	71.383 1		689.389
Niacinamide_SG1 31_MSC_750-245		Fail	1.38E+08
Procaine		Fail	4.11E+06
Tetracaine		Fail	2.41E+04
Benzocaine		Fail	9.78E+06
Boric_Acid		Fail	2.05E+04
Caffeine_SG1 31_MSC_550-2450		Fail	1.87E+05

Material Report

Date: 7/31/2009 1:46:22 PM Operator: Kirk M. Grates
Instrument:<unknown> Serial No.: 11005
Sample:Unk_45
Comments: Street Sample (Cocaine Base)

Constituent	ID	M-Distance
Cocaine Base	Fail	72.86

Confirmed by GC/MS

Material Report

Date: 7/31/2009 1:53:24 PM Operator: Kirk M Grates
Instrument:<unknown> Serial No.: 11005
Sample:UNK_940
Comments: Cocaine HCL

Constituent	ID	M-Distance
Cocaine HCL	Fail	410.184

Confirmed by FT/IR

Post-Evaluation Findings

Strengths/Results:

Strengths:

- Since the RxSpec 700Z utilizes a non-destructive analytical technique, there is no loss of sample to prevent further analysis of the sample using other technologies if required.
- This Vis/NIR instrument was noted to provide the user with relatively quick results within 20 to 30 seconds. Other portable or laboratory spectroscopy-based devices using techniques like Fourier Transform Infrared (FTIR) or Raman have been known to take several minutes or more to identify an unknown substance.
- This device can be utilized to identify fine powders, solids, and liquids.
- The unit is relatively lightweight and easily transportable to different locations.
- By having the ability to operate the instrument on either the internal battery or an external power source, the analyzer is not limited to power requirements.
- Dark-colored or fluorescent compounds are not a problem for this technology as they are for other spectroscopy-based techniques such as Raman.

Results:

- Chemometrics models developed by the evaluator as well as with assistance by a representative from ASD, Inc., “failed” to properly identify even standard samples. A pure boric acid standard was run, and the results produced by the RxSpec 700Z prompted the user with a “possible” ID for Cocaine HCl. A caffeine standard was also run, and it “Passed” as being identified as Cocaine Base.
- The evaluation was reduced in scope as a result of the aforementioned difficulties and resulted in excluding the sensitivity, reproducibility, portability, and environmental effects portions of the evaluation.
- If a chemometrics model is developed properly, it should account for most of the molecular variability occurring within a given collection of reference spectra for each of the compounds of interest, thus statistically reducing the occurrence of misidentification

errors. Unfortunately, the models created by both the evaluator and the ASD spectroscopist failed to meet adequate standards identification of these controlled substances, cutting agents, and non-controlled substances.

Areas for Improvement:

- Although the analyzer is housed in a protective aluminum briefcase, it is still vulnerable to extreme weather and harsh environmental conditions, thus requiring a secondary Pelican-type case for safe transport into and out of field locations. If the RxSpec 700Z was just encased in a Pelican-type case only, the unit would be much more ruggedized for transport and environmentally resistant to harsher conditions.
- Currently, the sample must be in close contact with the sampling window for analysis to occur. At this time, the evaluator is not aware of any option for this unit to have point-and-shoot capability. It is of the evaluator's opinion that an attachable fiber-optic probe would be a beneficial option to users requiring any standoff analysis applications.
- Per manufacturer recommendations, a daily USP test is to be conducted using seven certified Gray Scale Standards (pucks) from Avian Technologies to verify wavelength accuracy, photometric linearity, and spectrophotometric noise levels. Although this process does not take the user a long time to perform, the test should still be automatically conducted within the instrument and should not require user interaction. The warm-up time requirements as set by the manufacturer before running a USP test is 60 minutes for the instrument and a minimum of 15 minutes for the lamp.
- The minimum warm-up time requirement is at least 10 minutes prior to scanning samples. This time requirement seems to be excessive for a portable instrument and should be considered a burden to the unit's DC battery when AC power is unavailable.
- The analyzer should also use an automatic internal white reference standard when determining baseline measurements between sample measurements, instead of requiring the user to place the white reference onto the sampling window.

Limitations of Technology:

- Users may find that advanced knowledge of spectroscopy is necessary to optimize chemometrics model(s) to meet specific requirements for the detection and identification of controlled substances or any other compound of interest.
- Like any spectroscopy-based technology, mixtures can be problematic if the major component(s) are fillers, binders, diluents, excipients, APAP, etc., and the compound(s) of interest are minor component(s) at low concentrations. For example, the NIR analysis of a crushed 2.0-mg Xanax® would most likely not be able to identify the benzodiazepine alprazolam, a Schedule IV drug, present within the tablet. Only the fillers, binders, diluents, and/or excipients may be seen. A crushed 5-mg oxycodone/500-mg APAP tablet would also present some difficulty. The NIR would identify the APAP and maybe a few binders or fillers, but not the Schedule II drug oxycodone.
- NIR spectroscopy has limited specificity compared to other spectroscopy-based technologies due to its narrow scan range.

Training Requirements:

- Very limited training is needed to operate this instrument. The operator would only need to know how to collect samples into clean scintillation vials, select appropriate model(s) within the ASD Indico® Pro software, perform background correction measurements, and operate the graphical user interface (GUI) to run samples.
- It is of the evaluator's opinion that only an experienced spectroscopist can develop an effective chemometrics model(s) for use on the RxSpec 700Z, due to the complexity of both the GRAMS/AI and ASD Indico Pro software.

Health and Safety Issues:

- There were no noteworthy safety issues concerning the operation of this instrument.
- This unit can be considered safe to use if personnel are properly trained.

This project was supported by Award No. 2008-MU-MU-K003 for the Forensic Technologies Center of Excellence, awarded by the National Institute of Justice, Office of Justice Programs, U.S. Department of Justice. The opinions, findings and conclusions or recommendations expressed in this publication/ program/exhibition are those of the author(s) and do not necessarily reflect those of the National Institute of Justice.