

Alaska Scientific Crime Detection Laboratory

Controlled Substances Working Instructions

Issued: 10/26/2013
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Section 1

Carrier Gas Information

All of the gas chromatographs use UHP Grade (99.999%) helium as their carrier gas. Compressed gas cylinders are on site at all times. Two cylinders are connected to the gas manifold in Gas closet # 2124 that delivers gas to the instruments and extra cylinders are located in the compressed Gas Cylinder Room # 1175 located on the first floor of the laboratory.

The gas manifold system will sound an alarm when a cylinder in use drops to about 500 psi. Only the cylinder in use should have the valves open on the cylinder and that side of the manifold. Manually turn off the cylinder being taken out of service and close that side of the manifold. Update the magnetic indicator signs on the cylinders as appropriate (in use, empty, full). Ensure a full cylinder is available in the Gas closet for the next use.

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Section 2

GC-MS Instrument Parameters

Below is a table listing the GC parameters for the SCREEN.M method used in routine casework:

Injector Parameters	
Solvent A	Methanol
Solvent B	Hexane
Preinjection Washes	2 Solvent A, 2 Solvent B
Sample Pumps	3
Postinjection Washes	2 Solvent A, 2 Solvent B
Injection Volume (uL)	1
Inlet Parameters	
Mode	Split
Split Ratio	40:1
Inlet Temperature (C)	250
Septum Purge Flow (mL/min)	3
Column Flow (mL/min)	1
Oven Parameters	
Initial Oven Temperature (C)	100
Initial Oven Time (min)	1
Oven Temperature Ramp (C/min)	25
Final Oven Temperature (C)	325
Final Oven Time (min)	5
Run Time (min)	15

The instrument's software is programmed to automatically integrate detected peaks and, if a mass selective detector is being used, perform library searches of their mass spectra. The table below lists the integration parameters used by the SCREEN.M method.

Integration Parameters	
Integrator	RTE
Parameters File	screen.p
Data Point Sampling	1
Smoothing	Checked
Start Threshold	0.200
Stop Threshold	0.000
Baseline Reset (# points)>	5
If leading or trailing edge <	100.0%, Baseline drop else tangent
Minimum Peak Area	50000
Peak Location	Top
Maximum Number of Peaks	100

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Below is a table listing the MS parameters for the SCREEN.M method used in routine casework:

Mass Spectrometer Parameters	
Tune File	stune.u
EMV Mode	Gain (25)
Acquisition Mode	Scan
Low Mass	40
High Mass	550
Threshold	750
Sample #	2
Transfer Line Temperature (C)	270
Source Temperature (C)	230
Quad Temperature (C)	150
Standard Tune Parameters	
Tune File	stune.u
Tune Mass 1	69
Tune Mass 2	219
Tune Mass 3	502
Mass 50 Target (0.3 - 5%)	1
Mass 131 Target (20 - 120%)	55
Mass 219 Target (20 - 120%)	45
Mass 414 Target (0.3 - 10%)	3.5
Mass 502 Target (0.3 - 10%)	2.5
69 Abund. Target, counts (1E5 - 2E6)	500000
Peak Width Target, amu (0.4 - 0.8)	0.55
Maximum Repeller (10 - 42.84)	30
Emission Current (10 - 300)	34.6
Maximum Ion Focus (40 - 127.5)	90
Ion for Ion Focus Maximum (1, 2, 3)	3
Ion for Repeller Maximum (1, 2, 3)	2

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Section 3

GC Sequence Table

Acquired data will initially be saved to the following folder on each instrument:

D:\Instrument Name\DATA\RUNDATA

After the analyst has completed their review of the acquired data it will be moved from the RUNDATA folder to the following folder:

D:\Instrument Name\DATA\YYYYMM

At the beginning of each month, a new folder will be created for that month's data to be saved. Data folders that are over 1 year old will be deleted from the instrument computers.

Data file names will be the case number (including the year) followed by a differentiating character or characters if multiple samples from the same case are being analyzed. Data files for negative controls will be written in the same format with the prefix "B". When drug standards are analyzed, the file name will indicate what drug is tested.

The sample field will include the lab and item number of what is being tested and will indicate "blank" if the sample is a negative control. When analyzing a drug standard, the sample field will contain the drug name and its standard number.

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Section 4

FTIR and ATR Operating Procedures - Nicolet *iS10*

Polystyrene Analysis

- Display **Main Menu** of OMNIC software
- Install **Smart Omni-Transmission** accessory
- Select: **Collect, Experiment Set-up, Diagnostic, Align & wait, OK**

Note: IF the **System Status** shield is **RED**, go to “**System Suitability & Performance Verification**”

Note: If the **System Status** shield is **GREEN**

1. Insert the **Smart Golden Gate ATR** accessory, allow self-checks to occur
2. The **System Status** shield should remain **GREEN** (if **RED**, follow as before)
3. Collect Background (with the anvil up) picking the 2nd icon on the **Main Menu** bar
4. Collect Sample in Absorbance with the Polystyrene Standard (use only the metal framed standard with the label “**ATR STD**”, facing up)
5. **Find Peaks**
6. Click on **1600 cm⁻¹**, (this & all peaks above this peak will be labeled with their frequencies)
7. **Replace** (updates the display window, located in upper right corner, below the **GREEN** shield)
8. **Search** (a library match value of $\geq 90\%$ is an acceptable library match)
9. **Modify Display**, change **List of Matches** to “**1**”, **OK**
10. **Add NB**, fill-in comments & titles (adds a spectrum to notebook) ensure the spectrum is placed into **iS10 Polystyrene.NBK** folder
11. **View NB** (view notebook contents)
12. Examine **iS10 Polystyrene.NBK** (verify the report information, and that **1600 cm⁻¹** is labeled)
13. **Print** report and place in log book, **Close**

Blank Analysis

Note: Before analysis, install the ATR, **GREEN** shield displayed; acquire a “**Blank**” spectrum, then a “**Sample**” spectrum

1. A “**Blank Sample**” spectrum is obtained by tightening the anvil down onto the empty & clean diamond
2. **Col Smp** (Collect Sample data, **not Col Bkg**), when prompted, label case #, item #, etc.
3. **Search** (a library match value of approximately **80%** is usually acceptable, & if not both the anvil (sapphire) and the diamond crystal face must be cleaned)
4. If the “**Blank Sample**” is satisfactory, choose **Modify Display**, and set the “List of Matches” to “**1**”
5. **Add NB** (ensure in correct notebook with needed information), **Close**, and **Close** to exit

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Sample Analysis

1. A "**Sample**" spectrum is obtained by placing a sample onto the diamond & tightening the anvil down
2. **Col Smp** (Collect Sample data, **not Col Bkg**), when prompted, label case #, item #, etc.
3. **Search** (a library match value of approximately **80%** is usually acceptable)
4. If the "**Sample**" spectrum is satisfactory, choose **Modify Display**, and set the "List of Matches" to "**1**" or larger to show other library matches
5. **Add NB** (ensure in correct notebook with needed information), **Close**, and **Close** to exit

LIMS

Once all "**Blank Samples**" and "**Samples**" are obtained, go to **View NB** with the acquired data and print to the appropriate case in LIMS.

System Suitability & Performance Verification

If the **iS10** displays a **RED System Status** shield, (upper right corner), the operating parameters are out of specifications or the monthly scheduled update needs to be performed.

The following steps are necessary to obtain a **GREEN System Status** shield.

Install the *Smart Omni Transmission* accessory:

View, Configure System Status

1. **System Suitability** (Transmission accessory), **Configure, Collect**
2. Once a new reference sample is obtained, **Save, OK, OK** (to leave System Status Overview)
3. Click on **RED System Status** shield, **System Suitability, Run**
4. After the software completes all operations, a report is produced; if the system "**fails**", the **RED System Suitability** shield will display and Nicolet Technical Support needs to be contacted
5. If the system "passes", the **GREEN System Suitability** shield will display, a **System Suitability** report appears on the screen, **Print** and place in log book, continue onto **Performance Verification**

1. **Performance Verification** (Transmission accessory), **Run**
2. After the software completes all operations, a report is produced; if the system "fails", the **RED System Status** shield will display and Nicolet Technical Support needs to be contacted
3. If the system "passes", the **GREEN Performance Verification** shield will display, a **Performance Verification** report appears on the screen, **Print** and place in log book.

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ATR System Suitability with the **GREEN** System Status displayed:

1. Install the ATR, allow all self-tests to complete, **OK**
 2. With the ATR anvil up, **System Status** shield, **System Suitability** (ATR)
 3. The software will start operations and prompt for the "reference sample" (**only** use the Polystyrene Standard with the metal frame and with the label "**ATR STD**" facing up)
 4. If the system "**fails**", the **RED** System Suitability shield will display. Check the report to see if the test values (i.e., Y-Axis Accuracy, etc.) need correcting, correct problem, redo System Suitability. If test continues to fail, contact Nicolet Technical Support
- Return to **Main Menu** OMNIC software and begin Blank and Sample Analysis (at beginning of this section)

Note: **DO NOT** perform a **Performance Verification** with the ATR accessory, it will improperly make the system fail and the **RED** Status shield will display !!

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Section 5

Maintenance

Hydrogen Generator Maintenance

The hydrogen generator manufacturer also recommends preventative maintenance. Below is a copy of the suggested preventative maintenance schedule from the manufacturer's hydrogen generator service guide.

Description of Service Requirement	Recommended Service Interval			
	Daily	Weekly	6 Monthly	24 Monthly
Operation				
Check POWER ON indicator is illuminated	C	-	-	-
Check STATUS/FAULT indicators located on control panel	C	-	-	-
Check Water Level	C	-	-	-
Check Water Quality (Conductivity)	C	-	-	-
Check Desiccant Cartridge	-	C	-	-
6 Month Service Kit	-	-	R	-
24 Month Service Kit	-	-	-	R

Key:- C = Check R = Replace

A breakdown of the 6 and 24 month services from the manufacturer's service guide is below.

Item	Refer to Section	6 Month	24 Month
Change Water Filter	2.3.4	X	X
Change Environmental Filter	2.3.5	X	X
Change De-ioniser Cartridge	2.3.6	X	X
Change Water Pump	2.3.8		X
Change Float	2.3.9		X
Check rear fans		X	X
Check power supply fan		X	X
Check float operation		X	X
Check Cell Voltage			X
Check Desiccant Cartridge <i>(Change as required)</i>	2.3.2	X	X

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Procedures for performing these services can be found in the manufacturer's service manual. All 6 and 24 month services performed on the hydrogen generators will be recorded in their respective maintenance logs.

GC Maintenance

Assessing the chromatographic performance of the QC Mixture is the primary way in which the controlled substances discipline determines whether GC maintenance is necessary. However, the following preventative maintenance tasks will be performed at the minimum frequencies outlined below:

<u>Service</u>	<u>Minimum Frequency</u>
Liner Replacement	Every 2 Months
Septum Replacement	Every 2 Months
Gold Seal Replacement	Annually
Syringe Replacement	Annually
Column Trim/Replacement	Annually

Procedures for performing these services can be found in the instrument manufacturer manuals.

GC/MS Maintenance

Assessing the results of the standard tune is the primary way in which the controlled substances discipline determines whether MS maintenance is necessary. However, the following preventative maintenance tasks will be performed at the minimum frequencies outlined below:

<u>Service</u>	<u>Minimum Frequency</u>
Rough Pump Oil Change	Every 6 months
Ion Source Cleaning	Annually
Check PFTBA Level	Annually

Procedures for performing these services can be found in the instrument manufacturer manuals.

FTIR Maintenance

- Record all maintenance in the instrument logbook kept by the FTIR in the instrument room.
- Routine maintenance performed as needed:
 - Desiccant replacement
 - Cleaning of mirrors (service personal only)

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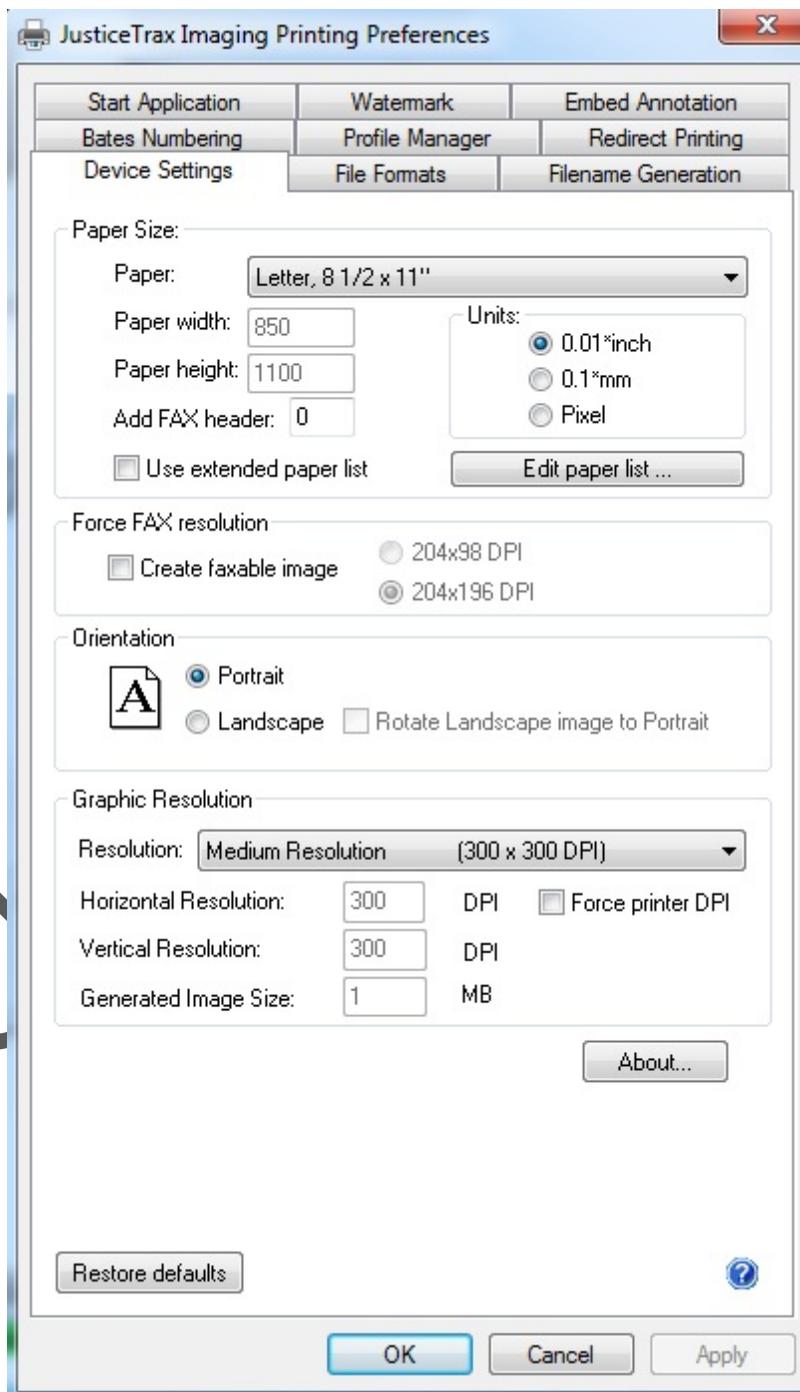
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Section 6

Justice Trax Print Instructions

The following three screen shots show the proper set up for printing to the imager on the instrument computers.

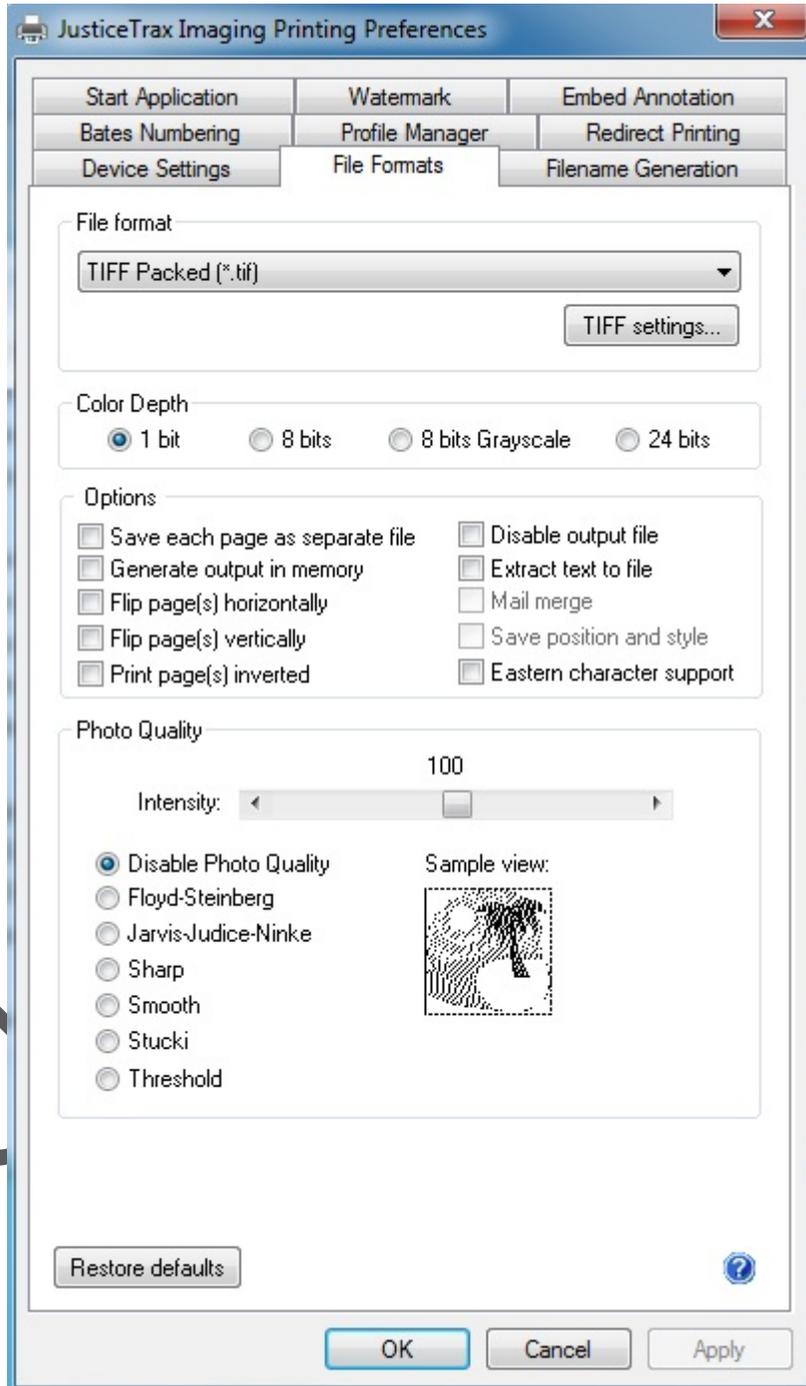


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JusticeTrax Imaging Printing Preferences

Start Application	Watermark	Embed Annotation
Bates Numbering	Profile Manager	Redirect Printing
Device Settings	File Formats	Filename Generation

Filename Generation Method

Enable Save As Option

Use the document name

Filename

Enter the full path and filename of the INI file:

...

Edit

Filename Prefix:

Filename Extension:

Use custom extension

Combine/Merge multiple documents into a single file

Using the 'Exact Filename' or 'Use the document name' option in conjunction with the 'Combine/Merge...' checkbox will allow multiple print jobs (documents) to be merged together. When this option is turned on, the printer driver will append the new pages to the existing file, instead of overwriting the file.

Output Directory:

C:\Users\Admin\Desktop\JT

...

Groupfile Options

Disable Group File Delete Group File After Printing

SharePoint®

Upload to SharePoint® Edit SharePoint® settings

Restore defaults ?

OK Cancel Apply

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Controlled Substances Key Control Policy

Each analyst is assigned the evidence locker in their work area. Any unassigned work areas' evidence locker can be used as needed. When not in use the key is stored in the lock indicating it is available for use.

The evidence lockers in the evidence room #2115 are unassigned and available for use as needed. When not in use the key is stored in the lock indicating it is available for use.

The duplicate key from each locker within the controlled substances laboratory is sealed in a labeled envelope. These sealed envelopes are locked in the primary controlled substances cabinet in the reagent prep room # 2116. These keys can only be opened by the discipline supervisor or designee.

If a locker key is lost, the discipline supervisor must be notified immediately.

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Appendix I

Revision History

2013 R2	2013 R1	Location	Revision made
5	5	Section 3	<p>The following was deleted.</p> <p>The comment field will include the analyst's initials. The analyst may include additional information in the comment field at their discretion. The operator name for all sequences will be designated as SCDL.</p>

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