Introduction

The Adsorbent Tube Injector System (ATIS) is a sample preparation device that provides a quick and efficient way to transfer calibration standards or samples onto an adsorbent tube. The ATIS employs the technique of flash vaporization to vaporize the sample in a continuous flow of inert gas. The inert gas carries the sample to the tube. After enough time has elapsed, typically less than 5 minutes, the tube is removed from the ATIS and analyzed using the appropriate technique for that adsorbent tube.

The ATIS performs these tasks:

- Injects calibration standards onto adsorbent tubes to calibrate your analytical system.
- Injects surrogates and system monitoring compounds onto an adsorbent tube before or after sampling.
- Removes moisture from the tube prior to analysis (dry purging).
- Vaporizes neat compounds into a gas-sampling bag to create a calibration standard.

The ATIS can be expanded:

- Thermally extract solid samples and collect the volatiles onto an adsorbent tube. The temperature range is adjustable from ambient to 150°C. Extraction glassware is available (ordered separately) that will accommodate solid samples up to 76mm (3”) length, and 13mm (1/2”) in diameter.
- Purge volatiles from an aqueous sample onto an adsorbent tube at ambient temperatures. A purge & trap / humidifier module is available (ordered separately) that interfaces with the ATIS. This module can also be used to generate a dynamic humidified stream of an inert gas for spiking calibration standards. The purge & trap module includes purge and trap glassware, and a separate flow controller that allows the user to set a separate purge (wet) flow rate independently of the (dry) flow rate.

How the ATIS Works

The ATIS is comprised of the following major components:

- Injection Glassware
- Adsorbent Injector Heating Block
- Carrier Gas Flow Control

Injection Glassware

The sample pathway of the ATIS is constructed of glass and stainless steel. The calibration standard is injected by a syringe through a replaceable septum in the center of the injection glassware. The injection glassware has an internal volume of 10 milliliters. This provides adequate volume for the vaporized liquid to expand.

Injector Heater Block

The injector heater block transfers heat produced by the block heater to the injection glassware. The heated glassware vaporizes the sample. A thermometer port is located in the top of the injector block to monitor the temperature during use.

Carrier Gas Flow Control

A supply of clean gas (nitrogen, helium, or air) is required to flow through the injection glassware during sample introduction. The gas carries the calibration standards / sample to the adsorbent tube during sample preparation.

The ATIS includes an adjustable flow controller and an on/off control valve. The flow controller will maintain a constant flow of gas, regardless of changing downstream pressures i.e., an adsorbent tube. The flow range is adjustable from 0 to 110mL/min. This flow controller provides better accuracy and control of the flow rate than a rotameter or needle valve. The on/off valve allows the gas supply to be turned off when the unit is not in use. A gas purifier is included that
will remove trace levels of hydrocarbon contaminants from your gas stream. However, additional purification may be required if your gas supply has high contamination levels.

**Safety Information**

The TALBOYS® Dry Block Heater included with this product is the heat source for the ATIS. This product operates at temperatures high enough to cause serious burns to the skin. A heat shield is provided to reduce the risk of getting burned while the ATIS is in use. The temperature of the heat shield will be approximately half of the set block temperature.

**Danger:** Do not use the ATIS in the presence of flammable or combustible materials or explosive gases. Fire or explosion may result, causing death or injury. This device contains electrical components that may ignite such materials.

**Danger:** Do not connect the ATIS to any flammable gases i.e., hydrogen. This will create a fire and/or explosion hazard.

**Warning:** The ATIS should be placed in a fume hood or have its own ventilation duct when in use.

**Warning:** Be careful when working around the ATIS when the block heater temperature is turned on.

**Warning:** Do not block the exit port of the pressure relief valve. Doing so could cause pressure to build in the system that may cause the septum to blow out of the septum holder, or worse case, cause the glassware to explode. The pressure relief valve will open to relieve the gas pressure if it exceeds 6psig.

**Warning:** Before performing any maintenance, turn off the block heater, unplug the electrical cord, and allow sufficient time to let the block heater to cool down.

In addition to reading this manual, please read the operating manual included with the TALBOYS Dry Block Heater (follow the instructions for the Standard Dry Block Heater) before using the ATIS.

**Specifications**

**Dimensions**
Requires a 61mm x 61mm (2’ x 2’) space to house the assembled unit.

**Power Requirements for the TALBOYS Block Heater**
- Cat# 28520-U ATIS 120 VAC +/-10% 50/60 Hz, 110 Watts, 0.9 Amps
- Cat# 28521-U ATIS 240 VAC +/-10% 50/60 Hz, 110 Watts, 0.5 Amps

**Certifications for the TALBOYS Block Heaters**

**Safety Standards:**
- IEC 61010-1 Safety requirements for electrical equipment for measurement, control and laboratory use. Part I: General Requirements.
- IEC 61010-2-010 Part II: Particular requirements for laboratory equipment for the heating of materials.
- UL Std.No. 61010-1

**EMC standards:**
- EN61000-3-2 EN61000-3-3, EN61000-4-4 EN55022-A, EN61000-4-2, EN61000-4-3, EN61000-4-11, EN61000-4-6, EN45501

**Associated EU guidelines:**
- EMC directive 89/336/EEC, LVD directive 73/23/EEC

**Temperature Range with all the ATIS parts attached:**
Slightly above ambient to 150°C (302°F)

**Pneumatic Specifications:**
Maximum operating pressure 60psig (Suggested operating pressure 25-30psig)

**Gas Flow Range:**
0-110 mL/min at 25-30 psig
The ATIS Components

- Operation Manual
  1. Block Heater
  2. Injector Heater Block
  3. Gas Purifier
  4. Copper Tubing 1/8” OD
  5. Flow Controller and On/Off Valve & Pressure Relief
  6. Polypropylene Tubing 1/8” OD
  7. 1/4” to 1/8” Reducing Union
  8. Injection Glassware
  9. 1/4” Union with Thumbwheel
  10. Heat Shield
  11. Thermometer
  12. Luer/Hose-Barb Adapter

Spare Parts
SP1. 6mm Ferrules “Black”
SP2. 1/4” Ferrules “Orange”
SP3. Half-Hole Septa
SP4. Spare Relief Valve and 3/16” Ferrules
SP5. 1/4” PTFE Ferrules “White”
Installation

Site Preparation
Place the ATIS in a fume hood and away from any combustibles. The ATIS requires a regulated supply (25–30psig) of nitrogen, helium, or air. Nitrogen is your best choice because it’s dry, clean, and relatively inexpensive. The ATIS also requires an electrical supply 120 VAC or 240 VAC (depending on the unit you purchased). The Block Heater must be plugged into a grounded outlet. See the operation manual included with TALBOYS Dry Block Heater for additional information.

Tools Required for Assembly (not included)
- Small tubing cutter
- Knife or scissors
- 7/16” open-end wrench
- 1/2” open-end wrench
- 9/16” open-end wrench
- Flow meter (to set the gas flow rate)

Assembling the ATIS
To assemble the ATIS requires you to cut the supplied tubing to fit your workspace and to attach the tubing to the appropriate fittings. We have installed the appropriate ferrules in each of the fittings for your convenience. The fittings that connect to the ATIS glassware include a VESPEL® ferrule. Use caution when tightening the fittings to the glassware – very little force is needed to create a seal to the glassware. PTFE ferrules are also included if you prefer to use them instead of the “orange” VESPEL type ferrules.

For additional help in plumbing the ATIS see the pneumatic diagram in Appendix 1 of this manual.

The picture to the right illustrates what the ATIS should look like AFTER you have assembled it.

Place the injector heater block (with the open slot facing to your right) in the opening of the dry block heater.
Using a tubing cutter, cut a piece of **copper tubing** to connect the **gas purifier** to a regulated gas supply (user supplied) of nitrogen, helium, or compressed air. Pressure should be set to 25 to 30 psig after installation is complete.

Using a wrench, tighten the nut 3/4 of a turn, past fingertight.

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<th><strong>Danger:</strong> Do not plumb the ATIS to any flammable gases i.e., hydrogen. This will create a fire and/or explosion hazard.</th>
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Cut a second piece of **copper tubing** to connect the **outlet** of the **gas purifier** to the **inlet** of the **on/off valve** connected to the flow controller.

Using a wrench, tighten both of these nuts 3/4 of a turn, past fingertight.

**NOTE:** Position the flow controller so you have access to it during use.

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Connect the **1/4” to 1/8” reducing union** to the **injection glassware**. The **SUPELCO Logo** on the glassware should face you.

Using a wrench, gently tighten the larger nut 1/8 of a turn, past fingertight.

**NOTE:** Over-tightening will cause the glassware to break.

| ![Image](image3.png) |

Cut a piece of the white polypropylene tubing to connect the **outlet** of the flow controller to the union you attached to the Injection Glassware.

Allow extra slack in the tubing so the tubing will absorb the shock if the tubing is accidentally bumped. This helps to prevent the glassware from breaking during use.

Using a wrench, tighten both of these nuts 3/4 of a turn, past fingertight.

| ![Image](image4.png) |
Slide the injection glassware into the injector heater block until the stem of the injection glassware protrudes through the recess of the injector block.

Install the thumbwheel to the 1/4" union. See the next section to choose the correct size ferrule for the adsorbent tube you plan to use.

Install the 1/4" union over the stem of the glassware that protrudes through the recess.

Using a wrench, tighten the CENTER hex portion of the union 1/8 of a turn while holding the injection glassware by the septum holder.

The recess secures the union so you can attach your adsorbent tube by using only the thumbwheel. The recess also promotes heat to transfer to the union. This prevents the calibration standard/sample from condensing inside the union.

Place the heat shield over the injector heater block so the septum holder protrudes through the hole of the heat shield. The base of the heat shield will rest on the gray cover of the dry block heater.

Insert the thermometer through the hole of the heat shield. Note: The thermometer port is dual tapered and will accept both a normal thermometer, or a smaller diameter digital thermometer.

The ATIS is now completely assembled. Your set-up should look like the picture to the right.
How to Attach your Adsorbent Tubes

The thumbwheel will accept tubes with either 6mm or 1/4" by placing the appropriate ferrule in the thumbwheel. For odd size tubes use the Luer/Hose-Barb Adapter included. The pictures below illustrate some examples.

NOTE: While using the ATIS, the temperature of the adsorbent tube will be close to the ambient temperature of your lab. However if the adsorbent tube is attached to the thumbwheel for a long period, the heat from the injector block will transfer to the attached adsorbent tube. The rate at which this occurs is dependent on both the block heater temperature and whether the adsorbent tube is made of glass or stainless steel. The transfer of heat will be greater with stainless steel than glass tubes.

### Installing Thermal Desorption Tubes

For 6mm OD tubes, install a 6mm ferrule “black” in the thumbwheel.

For 1/4" OD tubes, install a 1/4" ferrule “orange” in the thumbwheel.

### Installing Solvent Desorption Tube

Use the Luer/Hose-Barb Adapter and a piece of flexible tubing (not supplied) to connect odd size adsorbent tubes. To attach the Adapter, install a 1/4" ferrule “orange” in the thumbwheel. The smooth section of the adapter goes into the thumbwheel.

*Caution: Make sure the flexible tubing you choose will withstand the temperatures you plan on using. The wrong tubing could melt or off-gas by-products if left attached for a long period of time.*

### Installing Adsorbent Tubes with Male Luer fittings

The inner diameter of the Luer/Hose Barb Adapter will accept adsorbent tubes that have a male luer fitting.
Installing Gas-Sampling Bags

Gas-sampling bags can be attached using the Luer/Hose-Barb Adapter and a piece of flexible tubing. If the stem of the bag is 3/16” in diameter you can use 3/16” ferrule (included SP4) in the thumbwheel in place of the adapter.

Setting the ATIS Parameters

There are two parameters you need set; one is the flow rate, the other is the block temperature.

Setting the Flow Rate (Carrier Gas)

Insert the type of adsorbent tube you plan to use in the thumbwheel of the ATIS, and gently tighten the thumbwheel. With the gas supply on, attach a flow meter to the opposite end of the adsorbent tube. Turn the on/off valve attached to the flow controller to the ON position. (The arrow on the knob should be pointing towards the flow controller when it is ON.) Adjust the gas flow rate using the black knob of the flow controller.

- To increase the flow rate -- turn the knob counter-clockwise.
- To decrease the flow rate -- turn the knob clockwise.

The flow controller has a 13-turn adjustment knob; each complete turn of the knob is approximately 8 to 10mL/min change in the flow rate. The flow range of the flow controller is 0 to 110mL/min at 25-30psig.

Caution: Do not over-tighten the flow controller knob in either direction, or you could damage the flow controller.

Setting the Temperature

The power switch is located on the right side of the block heater. When the switch is in the center position, the unit is OFF. Depressing the switch to the left engages the Low Heat range control. Depressing the switch to the right engages the High Heat range control.

The temperature range of the block heater with all the ATIS components attached:

- Low Heat Range is slightly above ambient to approx. 100°C (212°F)
- High Heat Range is approx. 75°C to 150°C (167°F to 302°F)
Maintenance

Suggested maintenance for the ATIS includes the following:

- Check the temperature setting daily.
- Check the flow rate daily.
- Replace the septum after 25 injections.
- Replace the injection glassware after the first sign of any deposits.
- Replace the gas purifier annually.

Replacing the Pressure Relief Valve

In the event that you need to replace the pressure relief valve, a spare valve is included with the ATIS.

To replace the valve:
1) Remove the nut that holds the relief valve.
   *Note: You may have to break the old relief valve to remove it from the nut.*
2) Place a new 3/16” ferrule in the fitting.
3) Start the nut on the threads of the fitting.
4) Insert the relief valve through the nut and into the ferrule.
5) Gently push the relief valve so it is flush with nut.
6) Tighten the nut 1/8 to 1/4 of a turn. Do not over-tighten.

Guidelines for Using the ATIS

To accurately quantify the concentration of a sample acquired with an adsorbent tube, the analytical system must first be calibrated. The ATIS simplifies this task. Below are a few starting parameters, some experimentation may be needed to optimize the conditions for your application.

Allow enough time for the block heater to equilibrate to the desired temperature before injecting your standards. You will also want to make sure you have gas flowing through your adsorbent tube, prior to injecting your standard so the compounds will be carried to the tube.

Injecting Gas Phase Standards

Gas phase standards are available in compressed gas cylinders, made up in gas sampling bags, or gas sampling bulbs.

- Set the block temperature between 60° to 75°C.
- Set the flow rate of the carrier gas to 50mL/min.
- Allow the adsorbent tube to remain attached for 2 to 5 minutes after injecting the gas mix with the carrier gas on.
- The delivery rate of the gas syringe should be less than 10mL/min.

Large syringe volumes of gas standards can be injected through the septum of the injection glassware, but the injection rate of the syringe plunger must be slow. The rate that you inject the gas mix will have an affect on the total flow rate passing through the adsorbent tube. For example: If you injected a 100mL syringe volume of a gas mix into the injection glassware, and it takes you 10 seconds to inject the entire 100mL volume, the flow rate going through the adsorbent tube for those 10 seconds is 600mL/min. That is too fast. There is a good chance that the compounds in the gas mix could breakthrough the adsorbent and not be retained. In general, the delivery rate of the syringe should be less than 10mL/min.
**Injecting Liquid Phase Standards**

Liquid standards are typically made up in a solvent such as: methanol, hexane, or carbon disulfide.

- Set the block temperature 10°C above the boiling point of the solvent.
- Set the flow rate of the carrier gas to 50mL/min
- Inject the liquid standard, then allow the tube to remain attached for 2 to 5 minutes with the carrier gas flowing through the tube.

Note: If the compounds in your liquid standard are larger than C\textsubscript{12}, set the block heater temperature to 100°C

Unlike gas standards there is a limit to how much volume you can inject. If too much liquid is injected, the liquid could flashback into the plumbing of the system. Injection volumes of liquid standards should be less than 25 microliters. The injection rate for liquid standards can be quicker than that of gas standards, since the displacement of microliter volumes will have minimum impact on the flow rate going through the adsorbent tube. It is important to allow enough time for the contents of the liquid standard to be transferred to the tube before removing the adsorbent tube. For example; if the flow rate of the carrier gas is set to 50mL/min, after two minutes the injection glassware will be purged 10 times. Ten exchanges are typically enough to completely purge all of the compounds from the injection glassware.

**Dry Purging**

When samples are taken in humid atmospheres, the adsorbent tube may retain some moisture. Moisture retention is dependent on the type of adsorbent used. In general, it is best to remove this moisture prior to analysis. The ATIS simplifies this task. Make sure the injection glassware is clean prior to performing this task.

- Set the block temperature to 50°C
- Set the flow rate of the carrier gas to 50mL/min.
- The total time/volume to leave the adsorbent tube attached will need to be determined experimentally.

The key to dry purging is to allow enough (dry) gas to pass through the tube and carry the moisture away. Too much dry purge can actually start to push the compounds of interest through the adsorbent (Breakthrough). Dry purge is usually referenced as a volume instead of time.

**Recommended Dry Purge Volumes for a few adsorbents**

Tenax\textsuperscript{®} and Carbotrap\textsuperscript{™} adsorbents usually need only 0.25 liters of dry purge (five minutes at 50mL/min).

Carbosieve\textsuperscript{®} and Carboxen\textsuperscript{™} adsorbents may need anywhere from 0.5 liters to 3 liters, depending on how much moisture was in the air during sampling.

**Gas-Sampling Bags**

The ATIS can be used to vaporize neat compound(s) prior to injecting them into the bag. Gas sampling bags are often used to make calibration standards. Simply install the gas-sampling bag in the thumbwheel, and the process of filling the bag and injecting the compound can happen at the same time. By volatizing the compounds with the ATIS, this allows you to use the contents of the bag immediately without having to let it equilibrate overnight.
Using the Extraction Glassware (ordered separately)

Two different styles of extraction glassware are available, one with a micro connector, the other with a ground joint. The style you choose is your preference; both have their advantages. Both styles include a glass frit at the outlet and a replaceable septum. The replaceable septum allows a surrogate or internal standard to be injected during the extraction process. The glass frit helps to prevent solids from passing through to the adsorbent tube during the extraction. Solid samples up to 13mm (1/2") OD and 76mm (3") in length can be inserted into both styles.

To use the extraction glassware, simply remove the injection glassware from the injector heater block and reinstall the fittings as described in the assembly instruction of injection glassware. See Appendix 2: ATIS Glassware Configurations for additional information.

Replacement Parts

Consumable Parts:
- 28526-U Injection Glassware
- 20668 Half-Hole Septa (6mm OD 9mm Long) pk/100
- 22445-U Gas Purifier
- 22393 6mm Ferrules M-2A “Black” pk/10
- 22320-U 1/4” Ferrules M-2 “Orange” pk/10
- 29024-U 1/4” Ferrules Teflon “White” pk/10
See the SUPELCO catalog for additional ferrules

Replacement Parts:
- 33315 Block Heater 110 VAC
- 33318-U Block Heater 220 VAC
- Z423394-1EA Thermometer
- 28529-U ATIS Thumbwheel (Does not include the union)
- 28525-U Luer/Hose-Barb Adapter
Accessories:
28522-U  Purge & Trap/Humidifier Module
28523-U  Extraction Glassware with Micro Connector
64700-U  Replacement 20/20 Micro Connectors pk/6
28524-U  Extraction Glassware with 19/22 Ground Joint
64763  Replacement Clip for 19/22 Ground Joint pk/5
64762  Teflon Sleeves for 19/22 Ground Joint (used to create a tighter seal)
22754-U  Digital Timer
22435-U  Stainless Steel Tweezers

Troubleshooting

Below are some general troubleshooting tips. For expert answers to your questions about the ATIS, contact our Technical Service Department. (Phone 800-359-3041 or 814-359-3041, Fax 800-359-3044 or 814-359-5468), E-mail techservice@sial.com.

No flow is observed at the outlet of the adsorbent tube.
• Check to see if there is a blockage in the adsorbent tube.
• Check that gas delivery pressure is set to 25-30psig
• Make sure the on/off valve is in the ON position.
• Check for leaks; Check all of the gas connections for leaks.
• Replace the septum.
• Make sure the correct size ferrule is installed in the thumbwheel for the size of tube you are using.
• The pressure relief valve is open. If the backpressure of the adsorbent tube is greater than 6 psig the pressure relief valve will open, thus all or most of flow will exit the relief valve.

The flow controller will not maintain the set flow rate:
• Check that the gas delivery pressure is set at 25-30psig.
• There is an extreme difference in the backpressure of the adsorbent tube you are using than with the one you used to calibrate the flow rate. You should check the flow rate when using different types of tubes.
• The backpressure of the adsorbent tube exceeds 6psig and the pressure relief valve has opened.

I do not see any peaks after injecting my calibration mix.
• The gas flow may have been OFF during the injection.
• The temperature of the block heater is too low or it was off.
• There may be leak. Replace the septum; Check the injection glassware for breakage.
• Check the pressure relief valve.

I am seeing extraneous peaks in my analyses.
• Check the glassware, it may be contaminated.
• Gas purifier may need to be changed.
• May need to install additional gas purification.
• The calibration standard or sample may have flashed back into the pneumatics of the ATIS. This may require you to purchase a new flow controller, on/off valve, and to replace the tubing. Contact our Technical Service for assistance in ordering replacement parts.
Questions & Answers

Q: Can I leave the unit turned ON for an extended period?
A: Yes, leaving the temperature of the block heater on, and a flow of gas going through the injection glassware will help to keep the glassware clean and ready for use. To save electrical energy: Utilize the feature of the two temperature ranges of the block heater: Set the low range temperature to around 50°C, and use the high range setting for the temperature you are using to inject your standards. When you are not using the ATIS for an extended period of time, just simply set the power switch to the low range.

Q: Can I use PTFE ferrules instead of the VESPEL ferrules to connect the ATIS glassware?
A: Yes, You can use PTFE (TEFLON®) ferrules instead of VESPEL ferrules. The VESPEL ferrules were chosen for their inert properties and high reusability. All of the ATIS glassware uses 1/4” OD connections. However if you prefer PTFE ferrules we have included a pack of 3 with the unit.

Q: How can I be sure I completely transferred my calibration standard?
A: This may require you to do some experimentation by making several analytical runs of your spiked adsorbent tubes. Change the amount of time you leave the tube attached after making an injection from 2 to 5 minutes. Also, experiment by raising the temperature of the injection block. Compare the results. In most cases you will not need to change the flow rate from 50mL/min.

Q: How can I determine if my adsorbent tube is dry? (Dry Purge)
A: The best way is to weigh the adsorbent tube and record its tare weight prior to sampling. Then after sampling, reweigh the tube. If the difference is greater than 2 milligrams you may want to remove the moisture prior to analysis. Attach the tube to the ATIS and allow 0.5 to 1 Liter of the carrier gas to pass through the tube. Reweigh the tube. The adsorbent tube can be considered dry when it is within 1.0mg of the weight of the tube prior to sampling.

Q: Why does the pressure relief valve open at 6psig?
A: The pressure relief valve is designed to relieve the pressure if an adsorbent tube with a complete blockage was accidentally installed. The relief valve prevents pressure from building up in ATIS glassware. The pressure relief valve will reseal itself, once the pressure falls below 6psig.

Q: Does the white polypropylene tubing off-gas volatiles?
A: No, this tubing does not off-gas any volatiles at ambient temperatures. However if the tubing is heated, some volatiles should be expected. The maximum temperature of this tubing is 93°C (200°F).

Trademarks:
ATIS – Sigma-Aldrich
Carbotrap – Sigma-Aldrich
Carboxen – Sigma-Aldrich
TEFLON, VESPEL – E.I du Pont de Nemours & Co., Inc.
Tenax – Enka Research Institute Arhem
TALBOYS – Henry Troemner LLC
Appendix 1: ATIS Pneumatic Diagram

Suggested operating pressure to 25-30 psig
Maximum operating pressure 60 psig
(Regulator not included)

Gas Purifier

Copper Tubing

Regulator

Half-Hole Septa

SS Reducing Union 1/4" to 1/8"

SS Union 1/4" to 1/4" with Thumbwheel

Orange 1/4" M-2 Vespel Ferrule

Flow Element

Flow Controller

IN

OUT

Relief Valve

IN

Out

IN

White Polypropylene Tubing

Copper Tubing
Appendix 2: ATIS Glassware Configurations

1. Standard Injection Glassware Cat# 28526-U
2. Extraction Glassware with Ground Joint Cat# 28524-U
3. Extraction Glassware with Micro Connector Cat# 28523-U