

Thermo Electron
Smart Golden Gate™ MKII
Single Reflection ATR System



Sampling Notes

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THERMO ELECTRON SMART GOLDEN GATE MKII

SAMPLING NOTES

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1 Introduction

The Thermo Electron Smart Golden Gate MKII Single Reflection ATR system is a versatile accessory with a number of different sampling options for the quantitative and qualitative analysis of solids, liquids, pastes and microsamples.

The accessory uses a 2 mm x 2 mm square diamond, brazed into a tungsten carbide disc. The active area of the diamond is approximately 1mm diameter in the center.

The top clamp mechanism is supplied with two interchangeable compression head anvils. One of the anvils is fitted with a flat sapphire for compressing hard solid samples. The other anvil has a concave stainless steel surface for irregular samples (e.g. polymer pellets), preventing sample movement during compression.

When pressure is applied the anvil does not rotate against the sample. This ensures that heat is not generated by friction, as heat could change the sample. It also ensures that the sample is not displaced from the correct sampling position.

Corrosive Samples

The ATR element in the Golden Gate is high temperature bonded into tungsten carbide using a metal layer. Tungsten carbide and diamond have exceptional chemical resistance properties; However, we cannot guarantee the support disk or bonding material against corrosion from all materials, particularly under hostile conditions of elevated temperature.

We recommend that your sample is confined to the center of the diamond only when there is any risk of chemical attack upon the materials of construction.

A sample spot 2 microns deep with a diameter of 600 microns is sufficient. We recommend that a pipette, or similar glass capillary

Thermo Electron Smart Golden Gate MkII

tube, is used to apply small sample spots to the center of the diamond only.

The Golden Gate is capable of handling a diverse range of chemically aggressive materials. However, we recommend that in order to protect your accessory, your sample is cleaned off soon after analysis is complete.

For information on spares, consumables, and Golden Gate upgrade options please contact your local Thermo Electron office.

2. Using the Thermo Electron Smart Golden Gate MKII

General Principles

When using the Thermo Electron Smart Golden Gate MKII Single Reflection ATR accessory the sample is placed onto the crystal area (1) of the top plate. The contact between sample and crystal is improved using the clamp bridge (2) and an appropriate compression head anvil (3).

The diamond crystal is brazed into a surrounding support disc of tungsten carbide.

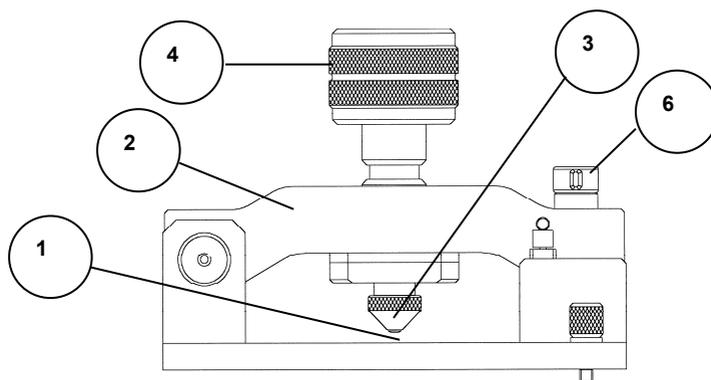


Fig 1: Side View of MKII Golden Diamond ATR Top Plate (Bridge Closed)

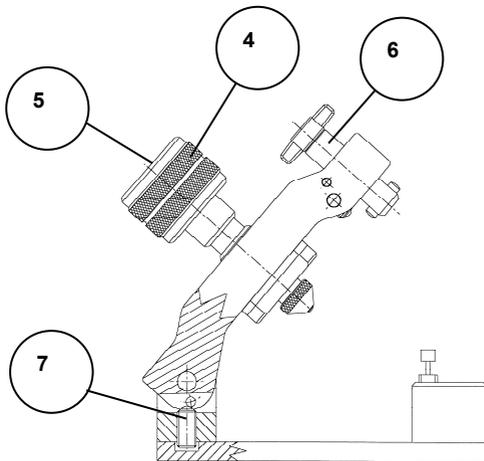


Fig 2: Side View of MKII Golden Gate Diamond ATR Top Plate (Bridge Open)

Features of the MKII Golden Gate Clamp Bridge

Torque Limiter Screw

The Thermo Electron Smart Golden Gate MKII Single Reflection ATR top plates are provided with a special built in torque limiter screw (4). When the torque limiter screw (4) is turned clockwise it will provide a maximum load of 80lbs (spread over the area of the anvil face) to the sample. After this load has been achieved the screw rotates without increasing the load. This means that there is no need to use a torque wrench for routine sampling. However, a torque wrench with torx head adaptor (10504 and 10505 respectively) can be supplied separately to provide loads up to 160lbs, using the torx head screw fixing (5) (See Fig 3).

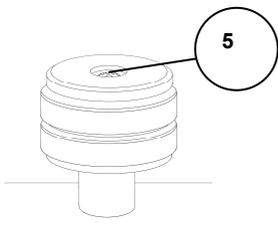


Figure 3: Torque limiter screw showing torx screw fixing

Quick Lock and Release Mechanism

The clamp bridge (2) has a quick lock and release mechanism (6). When pressure needs to be applied to a solid sample the bridge must be closed (see Fig 1). The quick lock and release screw is pushed down and turned clockwise 90° to lock the bridge closed. To release the bridge the screw is pushed down and turned anticlockwise 90°.

Important : *When closing the clamp bridge ensure that the anvil is retracted so that it will not crash down on the crystal or sample.*

Stay up Device

At the hinge point of the Golden Gate clamp there is a spring ball bearing device (7) that prevents an open bridge from accidentally falling back down to its closed position on the bridge support (8). When sampling liquids there is no need to use the bridge as the sample will be in intimate contact with the crystal. This device allows the bridge to stay open for convenience when sampling liquids (see Fig 2).

Preparing the Accessory for Analysis

For the Diamond ATR the active sampling area is about 1.0 mm diameter, in the center of the diamond. Samples smaller than this should be placed centrally on the diamond for optimum results.

2.1 Undo the bridge quick lock screw (6) and lift the clamp bridge (2) up past the stay up catch (7). The anvil (3) does not have to be in contact with the diamond (1) crystal face to record a background spectrum.

2.2 Ensure the diamond is clean and record a background spectrum.

Note: *If it is necessary to clamp the Sapphire Anvil (3) (10531 or 10522) directly onto the diamond without a sample, (e.g. to check for contamination) care should be taken to ensure that the anvil is level to the*

diamond crystal. This can be done by rotating the anvil (3) slightly before applying pressure. Only a small amount of pressure should be needed to check for contamination. It is not advisable to apply the maximum pressure loading without a sample.

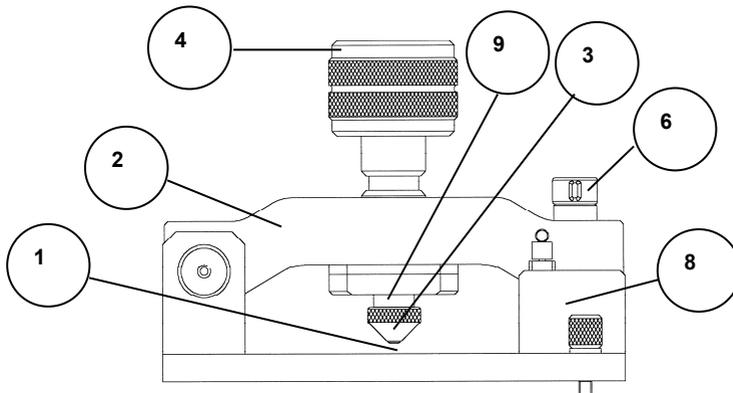


Fig 4: Closed Bridge MKII Golden Gate Diamond ATR Top Plate

Using for Different Sample Types

Important: *Golden Gate elements are selected for their freedom from natural structural flaws using optical test methods. It is possible to over-stress the diamond element by applying excessive point loads at the extreme corners.*

*If you suspect that your sample may contain exceptionally hard particles, ensure that only a small amount of sample is used and is placed centrally on the diamond. **Do not apply maximum loading immediately.** Use a torque wrench set to 20 cNm (approx 33 lbs). Acceptable spectra may be obtained without the requirement for the full load of 100 cNm (160 lbs).*

Powders

1. Prepare the accessory for analysis by collecting a background spectrum. (See 2.1 to 2.2, page 6).
2. Ensure the sapphire anvil (3) is firmly fitted to the end of the sample screw clamp (9) by pushing the hollow end over the retaining 'O' ring. Position the anvil to be parallel to the bridge.

Note: *When fitted, some movement of the sample compression head (3) is allowed to adjust to the sample shape during compression. This movement enables even pressure to be applied across the contact area.*

3. Apply the powder to the surface of the diamond (1).
4. Gently clamp down the bridge (2) using the bridge quick lock screw (6), ensuring that the anvil is clear of the sample.
5. Screw down the anvil (3) via the torque limiter screw (4). When a load of 80lbs is achieved the screw will rotate freely. The sample is now compressed between the diamond and sapphire anvil. Record the spectrum of the sample.
6. To remove or change the sample, release the pressure on the sample first by unscrewing the torque limiter screw (4) before undoing the bridge quick lock thumb screw (6). Rest the bridge up past the stay up catch (7).
7. Clean the powder from the diamond and wipe any powder off the sapphire anvil (3) of the compression head.

Flat Solid Samples

Flat solid samples are analyzed in the same way as powders, placing the sample face down on the diamond.

Pellets

Pellets are analyzed in the same way as powders, except that the pellet anvil is used. The pellet should be placed centrally on the diamond.

Ensure the conical recessed stainless steel pellet anvil (10532) (3) is firmly fitted to the end of the sample screw clamp (9) by pushing the hollow end over the retaining '0' ring.

Notes: *The conical recess head ensures that the sample is retained safely under the anvil during compression. Pellets of 1.5mm in diameter or less are not suitable for the recessed head. These are best analyzed with careful manipulation using the flat sapphire anvil.*

Fibers

Fibers are analyzed in the same way as powders. Lay the fiber centrally across the diamond crystal surface and tape down the ends out side the diamond crystal surface if necessary.

Note: *The fiber may be taped across a support frame and laid on the ATR top-plate with the sample centrally across the diamond. For larger wires or fibers the Wire Holder diamond ATR Top Plate (10565) can be used with an appropriate narrow or wide Grooved Anvil (10547) or (10548).*

Liquids and Pastes

1. Prepare the accessory for analysis. (See 2.1 to 2.2, page 6)

Note: *There is no need to use an anvil and the bridge clamp.
The bridge can remain open past the stay up catch (7).*

2. Apply a small quantity of the sample, sufficient to cover the diamond surface (only a thin covering is required).
3. If the liquid is volatile place the volatiles cover 10503 (Fig 5) over the sample to prevent evaporation.
4. Record the spectrum of the sample .
5. To remove the sample wipe the diamond and tungsten carbide surface with a cloth and suitable solvent.



Fig 5: Volatiles Cover (10503)

Pressure on Sample (Load)

The table on page 12 gives a guide to recommended loads for different sample types. The optimum load requirements for some typical samples are determined by recording spectra at increasing loads until the desired result is obtained. The optional Torque Wrench (10504) with Torx Adapter (10505), not supplied with the MKII Golden Gate, should be used for these settings (see Fig 6).

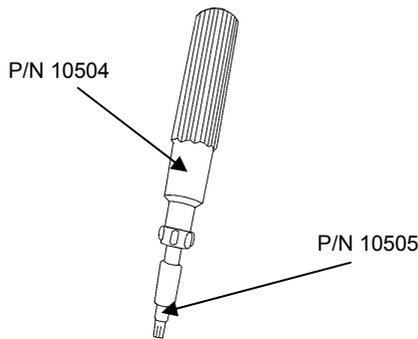


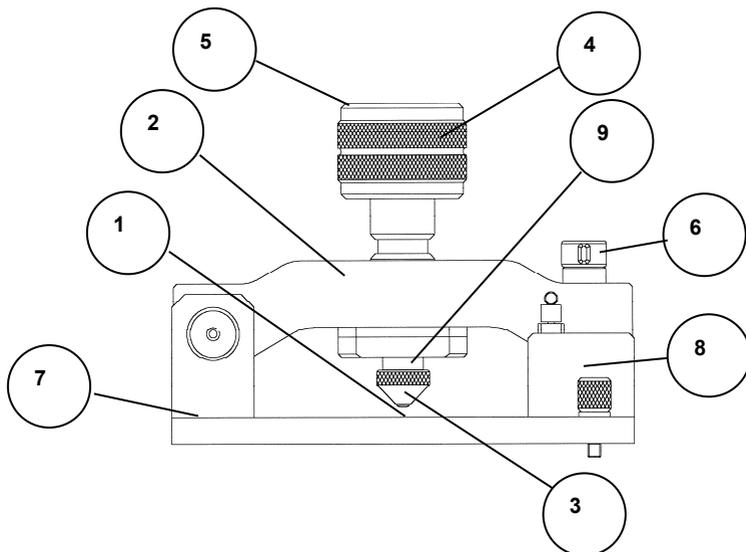
Fig 6: Optional Torque Wrench with Torx Head Adapter

Optional Torque Wrench

There is a torx screw fixing (5) inside the top of the torque limiter screw (4). The torx head adapter piece of the torque wrench fits in to this torx screw fixing in order to turn the torque limiter screw. Loads from 33 lbs up to 160 lbs (20 cNm to 100 cNm as shown on the torque wrench) can then be applied to the sample.

Type of Sample	Torque Setting (cNm)	Approx. Load (lbs)
Powders - soft	20 - 60	33 - 100
Powders - hard	40 - 80	67 - 133
Films and Plastic fragments	40 - 80	67 - 133
Polymer pellets - soft	20 - 60	33 - 100
Polymer pellets - hard	60 - 100	100 - 160
Fibers	40 - 80	67 - 133
Liquids and Pastes	Nil	Nil

3. Legend



- (1) Sampling Area/diamond Crystal
- (2) Golden Gate Clamp Bridge
- (3) Sampling Head/Anvil
- (4) Torque Limiter Screw
- (5) Torx Head Screw Fixing
- (6) Quick Lock Screw
- (7) Stay Up Device (internal fixing)
- (8) Golden Gate Bridge Support
- (9) Sample Clamp Screw Fixing

Worldwide Distribution

France

Eurolabo - Paris.
Tel.01 42 08 01 28
Fax 01 42 08 13 65
email: contact@eurolabo.fr

Germany

L.O.T. - Oriel GmbH & Co,
KG - Darmstadt
Tel: 06151 88060
Fax: 06151 880689
email:info@LOT-Oriel.de
Website: www.LOT-Oriel.com/de

Japan

Systems Engineering Inc. -Tokyo
Tel: 03 3946 4993
Fax: 03 3946 4983
email:systems-eng@systems-eng.co.jp
Website: www.systems-eng.co.jp

Spain

Teknokroma S.Coop C. Ltda
Barcelona
Tel: 93 674 8800
Fax: 93 675 2405
email: comercial@teknokroma.es

Switzerland

Portmann InstrumentsAG
Biel-Benken
Tel: 061 726 6555
Fax: 061 726 6550
email: info@portmann-instruments.ch
Website:www.portmann-instruments.ch

USA

SPECAC INC.
414 Commerce Drive
Suite 175,
Fort Washington,
PA 19034, USA
Tel: 215 793 4044
Fax: 215 793 4011

United Kingdom

Specac Ltd. - London
River House, 97 Cray Avenue,
Orpington
Kent BR5 4HE
Tel: +44 (0) 1689 873134
Fax: +44 (0) 1689 878527
Registered No. 1008689 England

Brilliant Spectroscopy™

www.specac.com

SPECAC INC.

414 Commerce Drive
Suite 175,
Fort Washington,
PA 19034, USA
Tel: 215 793 4044
Fax: 215 793 4011

SPECAC LTD.

River House, 97 Cray Avenue,
Orpington
Kent BR5 4HE
Tel: +44 (0) 1689 873134
Fax: +44 (0) 1689 878527
Registered No. 1008689 England