

# KBr Die Model 129

## Users Manual

P/N 0016-009 & 0016-001



P/N 700-0010

**Thermo** Spectra-Tech

Version 2.9

A Thermo Electron business



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## **General Information**

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### **The Manual**

This manual is designed as a tutorial to guide you through the installation of the KBr Die Model 129 and through a typical analysis. If you have any questions, please contact a Thermo Spectra-Tech Technical Representative.

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### **Packing & Unpacking**

The KBr Die Model 129 is shipped in a protective foam filled cardboard box. Upon arrival please check the box to ensure that all pieces have been received and that no pieces are damaged. Save the box for storage and shipment of the kits.

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### **Technical Support Center**

Technical materials describing the use and theory of attenuated total reflectance, diffuse reflectance and specular reflectance are available from Thermo Spectra-Tech. Additionally, a team of scientists is available at Thermo Spectra-Tech to answer any of your questions. If you encounter any problems or difficulties, or desire additional information please contact the Technical Support Center at 800-THE-FTIR.

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### **Contact Thermo Spectra-Tech**

230 Long Hill Cross Rd.

P.O. Box 869

Shelton, Connecticut 06484-0869

Phone: 203-926-8998, or 800-THE FTIR

Fax: 203-926-8909

[www.thermospectra-tech.com](http://www.thermospectra-tech.com)

[info@thermospectra-tech.com](mailto:info@thermospectra-tech.com)

## General Information

## Product Description

The KBr Die Model 129 produces high clarity discs of materials such as KBr (potassium bromide), KCl (potassium chloride), and CsI (cesium iodide). It can be used for pressing 13mm discs or for micro discs or pellets using the paper inserts. It is constructed of hardened stainless steel and aluminum. Its design and construction combine good performance, ease of use, maintenance and economy.

Discs will be produced starting at pressures of about 25,000 lbs. per square inch. Good clear discs will generally not be produced until the pressure reaches about 40,000 lbs. per square inch. *However, a disc does not have to be perfectly clear to obtain good spectral data.* This is true for KBr and KCl. The sample mat prevent a clear pellet from being formed.

When using KBr, approximately 300 to 400 mg will produce a 1mm thick/13mm diameter disc. The usual sample concentration is 1% in KBR or KCl to produce good spectra in the 4000 to 600  $\text{cm}^{-1}$  range. Beyond 600  $\text{cm}^{-1}$  concentrations of two to four times as much is required.

The Die can be used for pressing KBr, CsI and KCl. These matrix materials all have different densities, refractive indices and transmission ranges. The die cannot be used for pressing silver chloride and it cannot be used for flattening plastic pellets.

## General Information

### Material Properties

#### KBr

- Hygroscopic
- Transmission Range 40,000 - 400 cm<sup>-1</sup>
- Refractive Index 1.52 @ 1000 cm<sup>-1</sup>
- Most widely used matrix material.

#### KCl

- Hygroscopic
- Transmission range 40,000 - 500 cm<sup>-1</sup>,
- Refractive Index 1.45 @ 1000 cm<sup>-1</sup>,

#### CsI

- Hygroscopic
- Transmission range 40,000 - 200 cm<sup>-1</sup>,
- Refractive Index 1.74 @ 1000 cm<sup>-1</sup>

**Note:** Somewhat lower pressures are required for producing CsI discs than for KBr or KCl. Pellets or discs pressed from cesium iodide must be pressed a second and third time. This is accomplished by leaving the pellet in the die for five minutes or so, relieving the pressure of the press and bringing the pressure back to about 30,000 lbs. per square inch. The pellet is not removed from the die until after the third pressing.

### CAUTION

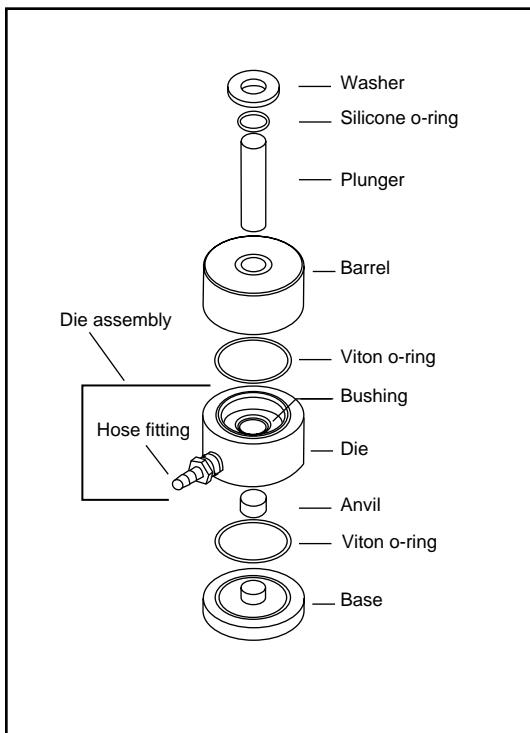
The base of the KBr Die must lie on the clean, flat platen of the press while pressure is applied. Failure to observe this precaution will result in cracking the base. The die must be placed in the center of the platen. DO NOT exceed 12,000 pounds total load on the gauge (60,000 psi at sample) when using the KBr Die Model 129. Total loads in excess of this limit may result in damage to die components.

# Operation

## Assembly & Sample Loading

### Press Requirements for the Die

- Minimum opening of at least 3 1/2".
- Capable of producing a total load of at least 12 tons.



### Assembly and Loading of Sample

#### *Step 1*

- Place the base on a flat surface and insert the "o-ring" in the groove.
- Place the die assembly over the post in the base.
- Insert the anvil POLISHED SIDE UP into the bushing of the die.

#### *Step 2*

- Load the prepared sample through the bushing hole and onto the anvil.
- Level the sample matrix material using a microspatula or a clean, dry, glass rod or shaking side to side.

#### *Step 3*

- Place the second "o-ring" in the groove and place the barrel on top.
- Insert the plunger and place the "o-ring" and washer around the plunger.

**Figure 1:** Parts and general assembly order.

# Operation

## Assembly & Sample Loading

### Pressing the Pellet

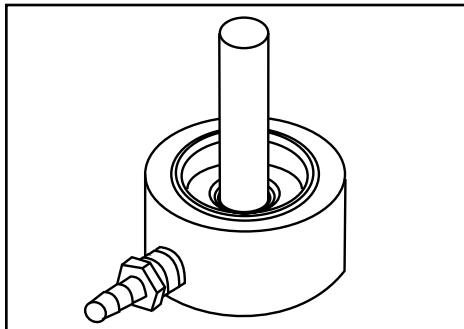
- Place the complete die in a hydraulic press and connect the vacuum line.
- After two minutes under vacuum, press the pellet.
- After an additional two minutes, release the pressure and the vacuum.
- Wait one minute before removing the die from the press.

Conversion for 13mm Die:

$$\text{lbs. total load} = .205 \times \text{psi}$$

For pressure	Use gauge reading
25,000 psi	5125 lbs. total load
40,000 psi	8200 lbs. total load

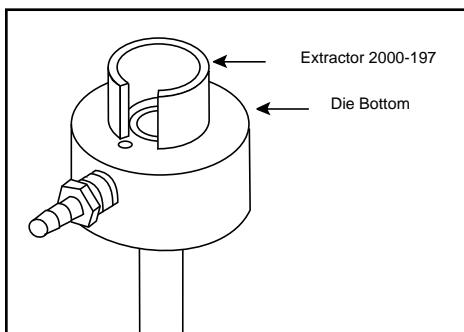
**Caution: DO NOT exceed 12,000 pounds total load on the gauge (60,000 psi at sample)**



### Ejecting the Pellet

After a pellet has been pressed, remove the base with the "o-ring", the barrel with "o-ring", and the washer with "o-ring". The anvil and pellet will still be in the die.

- Rotate the assembly upside down and Insert the die in the press.
- Place the extractor over the aperture for ejection of the pellet.
- While the press is being pumped use one hand to hold the assembly stable.
- As soon as the anvil clears the well, slow the pumping rate down.



After the disc is ejected, transfer it immediately to the magnetic holder (PN 0016-008) or suitable pellet holder and record the spectrum as quickly as possible to reduce the chance of absorption of water that may alter your spectra.

## Operation

## Cleaning and Storage

KBr is a very corrosive material which will rust the stainless steel parts if it is not completely removed. Please observe the following precautions when cleaning and storing the die:

- A nylon bottle brush can be used to scrub the well of the die.
- The components may be washed with a mild detergent and water.
- Avoid washing compounds containing trisodium phosphate.
- Rinse the inside die components with acetone and dry with compressed air.
- All die components should be stored in an air-tight desiccator when not in use.
- A light oil can be applied to the stainless steel parts to prevent rusting if the die is used infrequently.

**NOTE: All components should be disassembled and stored separately after cleaning.  
Trapped moisture may cause rusting.**

# Operation

## Paper Inserts

### Use of Paper Inserts for Micro Pellets

The paper inserts permit the use of much smaller amounts of matrix material and therefore smaller amounts of sample are required. Five types of paper inserts are supplied. All paper inserts are 13mm in diameter and 0.5mm thick.

Aperture size	Approximate amount of sample needed
1.5mm diameter*	20 mg
1.5 mm x 9mm	85 mg
3 mm diameter	45 mg
5 mm diameter	80 mg
11mm diameter	150 mg

#### For smaller size pellets:

- Assemble the die in the same manner as for 13mm discs.
- Place the insert on the polished surface of the anvil.
- Place the sample mixture into the aperture in a mound.
- *Do not smooth the preparation prior to pressing.*
- Press the pellet under vacuum in the same manner as a 13mm disc.

\*The use of a 1.5mm pellet is recommended only in conjunction with a beam condenser.

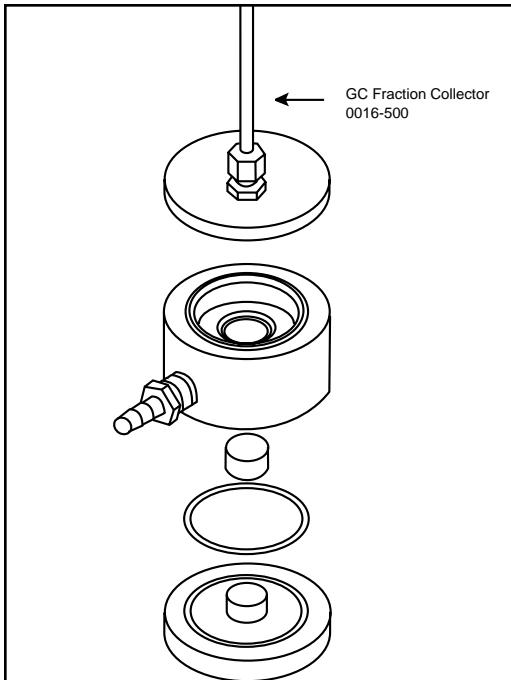
**Note:** *The force required to produce a good micro pellet is only one-fifth to one-fourth as much as it takes to produce a macro pellet.*

# Operation

## GC Fraction Collection

### GC Fraction Collection 0016-001 / 0016-500

The GC Fraction Collection assembly (PN 0016-001 / 0016-500) is used for the collection of fractions which are eluted at about 150°C or above. It is best suited for collecting compounds which condense as solids. However, many compounds are eluted as liquids. While liquids can be collected with the assembly, the preparation of a disc satisfactory for transmission measurements will be somewhat difficult. The capture of a liquid in the KBr causes the formation of a slush-like mixture in the well of the assembly. A disc pressed from this slush will invariably be opaque. Such a disc should be dried to remove excess liquids. It should be ground with a like amount of KBr powder and pressed again. Regrinding, diluting with KBr and pressing may have to be repeated several times.



The collection of compounds which are eluted as solids is simpler because these usually condense at very fine particles. Regrinding and pressing the disc prepared as a result of collection is sometimes necessary. Use of CsI is not recommended for GC fraction collection.

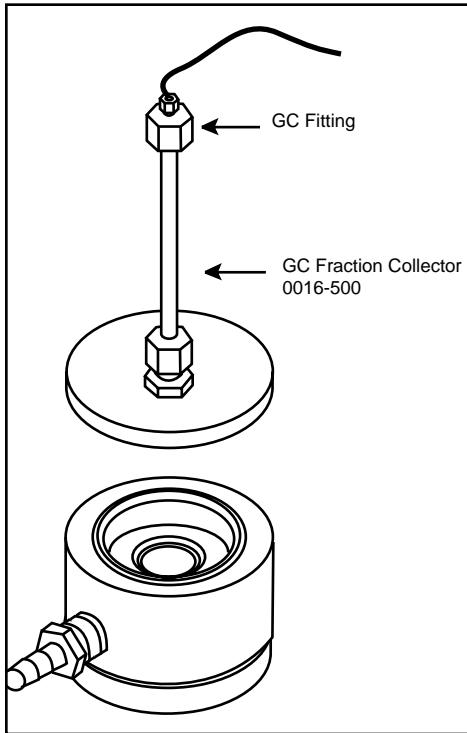
### KBr Die Assembly for Gas Chromatograph Fraction Collection

No fitting is supplied for connecting the die to the chromatograph. Various companies make a number of fittings suitable for this purpose. The choice of fittings depends on the diameter of the exit tube from the chromatograph. For example, the 1/8" diameter of the fraction collector can be joined to the 1/8" chromatographic outlet with the Swagelock brass union 200-6.

The tube which leads to the fraction collector assembly is supplied straight, but can be easily bent to meet individual requirements.

# Operation

## GC Fraction Collection



### KBr Die Assembly for Gas Chromatograph Fraction Collection

Attach the fraction collector assembly to the chromatograph outlet. Keep the tube which connects the chromatograph to the die at the same temperature as the column (this is accomplished by wrapping a heater tape or ribbon powered by a variable transformer). Assemble the lower portion of the die (see page 6, step 1).

Place 200mg of KBr over the anvil. The die and the fraction collector assembly are now ready for use.

As soon as the desired peak begins to appear on the recorder, complete the assembly by slipping the die portion quickly over the collection assembly.

Keep the collection assembly in place until the peak is completed. Then quickly remove the die section and finish assembling the die (see page 6, step 3). The disc must be pressed as quickly as possible.

## **Warranty**

### **Warranty**

#### **WARRANTY**

1. Subject to the conditions hereinafter contained, the Seller warrants that each item sold to the Buyer hereunder, shall be free from any defects in material and workmanship. This warranty extends to the original Buyer only and shall terminate six months from the date of delivery of the items sold hereunder. The Seller's obligation under this warranty is limited (i) to repairing, or if beyond repair, to replacing during the warranty period with reasonable promptness items returned to the Seller in accordance with paragraph 2 hereof and found by the Seller to be defective, or (ii) to returning, at the Seller's option, the purchase price to the Buyer. Seller shall not be liable for special or consequential damages to anyone. Except as stated above, the Seller makes no warranty either expressed or implied in fact or in law.
  
2. Items are not to be returned unless approved in writing by an authorized representative of the Seller. Transportation charges are to be prepared by the Buyer unless the Seller in such approval specifies otherwise. The Seller may, in its discretion, replace any or all returned items within a reasonable time after the Seller finally determines that the returned items are not in accordance herewith, and in such event, the Seller shall not be liable for any damages arising from the defective delivery or delay caused thereby.

## Accessories and Replacement Parts

### Accessories

#### KBr Die Model 129

<i>Part Number</i>	<i>Description</i>
0016-009	Macro - Micro KBr Die Model 129 Kit Includes; Anvil, Plunger, Base, Die, Barrel, "o-rings", Washer, Extruder and one dozen each of 1.5mm, 3mm, 5mm, 11mm diameter aperture, and 1.5 x 9mm rectangular paper aperture inserts and detailed instructions.
0016-001 Fraction	Macro-Micro KBr Die Model 129 with Gas Chromatograph Collector (includes all items from above)

#### Magnetic KBr Pellet Holder (PN 0016-008)

- Holds any macro 13mm potassium bromide pellet
- Universal slide holder
- Stainless steel backplate
- Two masks of soft permanently magnetized rubber (1-round, 1-oval)

#### KBr Pelletizing Accessories (PN 0016-002)

The micro funnel 0016-002 placed in the KBr Die Model 129 precisely positions the sample-matrix mixture (usually 0.02mg in 30mg) directly into the aperture of the micro paper inserts. The tamper directs the entire sample to the aperture, concentrates it and reduces any chance of loss of sample.

#### Micro and Macro Paper Inserts for KBr Die

<i>Part Number</i>	<i>Description</i>
0016-004	Paper inserts, 1.5mm diameter aperture, package of 200
0016-005	Paper inserts, 1.5 x 9mm rectangular aperture, pkg. of 200
0016-006	Paper inserts, 11mm diameter aperture, package of 200
0016-025	Paper inserts, 3mm diameter aperture, package of 200

## Accessories and Replacement Parts

### Replacement Parts

0016-026 Paper inserts, 5mm diameter aperture, package of 200

#### Replacement Parts for KBr Die Model 129

<i>Part Number</i>	<i>Description</i>
2000-188	Stainless steel base
2000-190	Stainless steel anvil
2000-191	Stainless steel plunger
2000-192	Aluminum washer
2000-193	Aluminum barrel
2000-197	Split Ring Extractor
0016-003	Replacement "O" Ring Kit, set of 3, (2 large, 1 small)
833-0000	Hose Fitting
0016-500	GC Fraction Collector Adaptor
0016-502	Die Assembly (Includes: die, bushing and hose fitting)

#### Micro KBr Pellet Funnel with Tamper

<i>Part Number</i>	<i>Description</i>
0016-002	Filling Funnel with Tamper
2000-200	Tamper only

#### Powders

<i>Part Number</i>	<i>Description</i>
0016-015	KBr Powder, 100 gm bottle
0016-030	KBr Powder Packets pkg of 25
0016-031	KBr Powder Packets pkg of 100
0016-032	KBr Powder Packets pkg of 250
0016-033	KBr Powder Packets pkg of 500
0016-020	CsI Powder, 50 gm bottle
0016-021	Kcl Powder, 25 gm bottle

#### Magnetic KBr Pellet Holder

<i>Part Number</i>	<i>Description</i>
0016-008	Magnetic KBr pellet holder



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*www.thermospectra-tech.com  
info@thermospectra-tech.com*

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Thermo Spectra-Tech  
230 Long Hill Cross Rd.  
PO Box 869  
Shelton, CT 06484-0869

Phone: 203-926-8998  
800-THE-FTIR  
FAX: 203-926-8909