

How do I prepare high quality KBr pellets?

Generally, it is easy to produce a good quality KBr pellet if an evacuable pellet die is used correctly. However, some faults in the produced sample pellet may occur due to a variety of reasons. Some of these faults and their remedies are tabulated below.

The faults described are for pure KBr or other halide salts which do not contaminate the sample. When the sample is added to the halide salt the clarity of the disc will depend to a large extent on the quantity and type of sample. Usually 0.1 to 2% weight to weight of sample to KBr is perfectly adequate. The overall quality of a pellet is largely dependent upon the quality of the KBr or halide salt powder used, which should always be of a spectroscopic grade of purity.

Potential KBr pellet errors and solutions		
Fault	Cause	Remedy
Pellet lacks clarity.	Sample damp, contaminated KBr powder or insufficient pressure has been applied.	Dry sample, change KBr or apply more pressure.
Pellet clear, but shows opaque spots.	Powder not uniformly flat, leaving larger particles which fail to vitrify when pressed.	Sieve powder to extract coarse grains, before re-grinding/re-pressing.
Pellet is cloudy.	Insufficient evacuation time or seal leaking.	Check die seals or lengthen evacuation time.
Pellet gradually becomes cloudy.	Damp powder or damp atmosphere.	Dry the KBr powder or sample, check seals on the die and lengthen evacuation period.

To ensure that a sample pellet is produced which will enable accurate spectra of samples to be obtained, it is essential that the sample is thoroughly blended with the halide salt powder. Blending can be achieved either by using a mortar and pestle (P/N GS03600) or by using a grinding mill such as the Specamill (P/N GS06000).

If a pellet breaks apart, then possibly not enough total mixture is being used in the die. Under compression the disc has become too thin and fragile. Excess moisture in a KBr disc could cause it to fragment and insufficient pressure or load on the sample when in the die can mean that the KBr/sample mixture has not been sufficiently compacted. For a 13mm diameter die with a total KBr/sample mixture mass of 400mgs, a load of 6 to 7 tons on the press gauge will produce a disc of approximately 1 to 1.5mm thickness.

Specac, River House, Cray Avenue, Kent, UK BR5 4HE