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THE KBr TECHNIQUE

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In a comparatively short time, the elegant method of sample preparation for infrared spectroscopy, concisely known as the KBr Technique, has become firmly established as a unique method for handling solid samples.

Until about 1951-52, infrared examination of solids was accomplished with solutions, mulls, or to a limited extent, with dry powder films. Solution techniques are fine provided a suitable solvent can be found. It must have a large enough "window" in the pertinent infrared region and relatively high solubilities are required to obtain good pictures. Mull techniques using mineral oil, hexachlorobutadiene or fluorinated oils, such as perfluorokerosene or fluorolubes, are excellent if the absorptions bands of the mulling agents do not interfere, if reflection losses can be tolerated and if strictly quantitative results are not needed. Dry powder films are useful, too, but unless the particle size is very small, the infrared radiations are scattered and the resultant pictures are poor. Films cut thinly by a microtome, rolled on a press or cast from solution also have their place, but how does one handle a recalcitrant solid of which there is only a very small amount, which is insoluble in any usable solvent and intractable to mulling, melting or casting as a thin film?

In the period 1951-52, Stimpson and O'Donnell (1) in this country and Schiedt and Reinwein (2) in Germany, used the technique of diluting materials in solid KBr and pressing the mix to give a fused disk which could be introduced into the

light beam of a spectrophotometer. (Cook and co-workers (3) reported developing the same technique during the period 1947-49 but their work was classified and hence not formally published.) The idea was quickly taken up and, although other halides have been tried (4, 5, 6 and 7), potassium bromide has remained the favorite for pressed disk work. In special instances there are advantages of other matrices. Teflon, with its complete chemical inertness, has been used successfully (38) but it has a strong band at 1000 cm^{-1} to 1500 cm^{-1} and so suffers from the same disadvantage as Nujol mulls and solvents. Hales and Kynaston (5) decided to use KCl because of its lower chemical reactivity and higher heat stability. They developed a technique of producing a pure finely divided KCl powder by precipitating the KCl from a saturated aqueous solution of KCl which is poured into ice-cold analytical grade HCl. The final product gave glass-clear disks and was used successfully for qualitative and quantitative work. The overall transmittance of KCl in the infrared is not as favorable as that for KBr, however.

The matrix must be finely ground and intimately mixed with the sample either by grinding in a mortar or mixing in a mill. Concentrations of sample in halide from around 0.01% to as high as 0.5% may be used. Disks weighing 100 to 400 mg commonly have 0.1-0.5% of sample. Too high a concentration causes opacity of the windows so it is better to increase pellet thickness rather than sample concentration. After grinding,

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the mix is pressed in an evacuated die at fairly high pressures forming a clear disk from which the infrared picture may be obtained. The spectrum usually shows little or no scattering of light above 2 microns and with KBr the unobscured picture is available as far out as 28 microns. Since pure KBr and KI are transparent in the ultraviolet as well as in the infrared, an additional bonus is obtained. Some general requirements are:

1. High quality KBr or other halide as a matrix material;
2. Grinding method capable of producing an optimum particle size;
3. An evacuable die capable of withstanding high pressures;
4. An efficient press.

Matrix Problems

Choice of the halide, its chemical purity and physical form are of great importance. KBr powder must have a small particle size to prevent loss of light by scattering and reflection, but not so small that the rate of water absorption will be too great. A number of workers have shown that a particle size below 10 microns is good, and even very much smaller particles are desirable, but these are often impractical to produce and difficult to work with because of extremely rapid water absorption.

Another physical property affecting the sharpness of spectra is the refractive index (8, 9). If the refractive indices of the medium and sample are closely matched, the best curve is produced. Otherwise asymmetrically shaped bands may occur. These are characteristic of the well-known Christiansen effect due to the rapid divergence in refractive index between the two. When the particle size is small enough, however, the effect often vanishes (10). Large differences in refractive index between matrix and sample may also shift the position of the band. In the spectrum of cobalt mercury thiocyanate the $\text{—C}\equiv\text{N}$ band moves from 2100 cm^{-1} in KI to 2150 cm^{-1} in NaCl.

Additional factors affecting the KBr technique are absorption of the sample by the matrix (11) and actual chemical reactions between matrix and sample (9, 15). Farmer (12) showed that the spectrum of benzoic acid changed when pressed in different alkali halides and then compared with a mull in Nujol. In KI and Nujol the spectrum of benzoic acid was similar to that of a dimer, whereas in NaCl the spectrum approached that of the monomeric form obtained in dioxane solution. The monomeric molecules are absorbed on the surface of the NaCl particles. The strong dimer band at 1294 cm^{-1} is displaced as we move from NaCl to KI, the latter showing the band at 1220 cm^{-1} . Tolk and van der Molen (13) showed that the benzoic acid spectrum changed with time. Immediately after pressing, the pellets gave spectra like Farmer's, but after a few days the dimeric form was visible again. Karagounis and Peters (14) obtained similar results to Farmer's but interpreted them as due to the polarization of the dimers absorbed on the surface by the ionic fields.

In extreme cases, such as after prolonged grinding, a sample may become hydrated or completely changed into a salt. Grinding and pressing, even of very short duration, can also produce polymorphic changes and distortion of the structure (18).

For example, benzoic acid may be completely changed to potassium benzoate by grinding for 20 hours (9). Here an exchange reaction takes place and, among other spectral changes, the typical carboxylic acid band at 1695 cm^{-1} moves into the ionized carboxyl or salt region between 1550 cm^{-1} and 1610 cm^{-1} . A further illustration is given by 2-aminonaphtha-

lene-7-sulfonic acid (9). For short periods of grinding the spectra in Nujol or halide are the same, but with continued grinding the spectrum changes and shows band splitting and frequency shifts as the exchange reaction and later progressive hydration of the molecule occur. Neuderte and Ropke (36) showed that hydrates may be formed during pressing, this being particularly so in the case of organic acids and salt where even the very low water content of the KBr is sufficient to form the hydrate. Not all hydratable compounds form hydrates during pressing, and although Dietrich (37) described a method for completely removing water from KBr, this is not done routinely and so care must be taken. Acid chlorides can also react with residual moisture giving bands due to the impurities formed by reaction with the water. Meta nitro-benzoyl chloride pressed in KBr shows a weak —COOH band at 1695 cm^{-1} which is not present in the Nujol mull.

Several authors have mentioned the formation of mixed crystals and complexes with halides (9, 16, 17, 19, 20). Ketelaar, Haas and van der Elksen noticed changes in the infrared spectra of bifluorides when pressed in alkali halides as compared with spectra run in Nujol. In the case of the three bifluorides of sodium, potassium and ammonium, the spectra in Nujol always gave two bands, one very broad, in the region $1200\text{--}1600\text{ cm}^{-1}$. In halides the picture differed. For potassium chloride, a pair of sharp bands occurred in the region $1200\text{--}1600\text{ cm}^{-1}$, shifted from those in Nujol but remarkably constant in frequency. This was interpreted as mixed-crystal formation. For the three bifluorides in sodium bromide, four bands occurred in the $1200\text{--}1600\text{ cm}^{-1}$ region, interpreted as partly mixed-crystal formation and partly reaction of the bifluoride with the halide, potassium bifluoride being partially reacted with the sodium bromide to give sodium bifluoride.

Duyckaerts (9) noticed a variation of the $\text{—C}\equiv\text{N}$ frequency when potassium cyanide was milled in Nujol or pressed in halides. Stewart (16) showed that the spectra of thiourea pressed in CsBr, KI and KBr differed from each other and, in turn, differed greatly from the spectra when pressed in NaCl, KCl, NaBr and NaI which were identical with a Nujol mull. A study of the spectra suggested that a foreign body was introduced into the thiourea lattice thus perturbing the molecular vibrations. This has been interpreted as mixed-crystal formation, and further evidence for this was given by Acampora, Tompa and Smith (17) who observed mixed-crystal formation when pressure was applied to mixed KCl-KBr powder.

When a mixture of two compounds is to be pressed into a KBr disk, their mutual reactivity must be considered. Nitrobenzaldehyde forms a hydrazone with 2:4 dinitrophenylhydrazine, and this actually occurs during pressing.

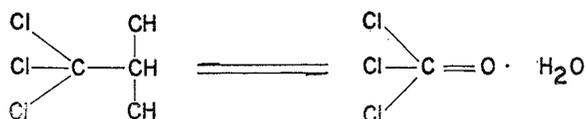
Consideration must also be given to labile substances such as azides which may well decompose during pressing. Even more stable compounds have been known to break down during disk preparation. The decomposition products can wreak havoc with interpretation unless the breakdown has been considered. Discoloration of the disk or a thoroughly diffuse non-specific spectrum often give this away. The converse situation is also true that in some cases where decomposition occurs in solution the KBr spectrum is fine.

Apart from reactions with the halide, the procedure for making pellets has also been shown to give rise to polymorphism and crystalline distortion. Baker (18) showed a distinct polymorphic change in alpha-naphthalene acetamide after a comparatively short grind with KI. He also showed that benzil, when ground for a long time, gave a spectrum similar to that of the substance dissolved in carbon disulfide, i.e. the benzil completely lost its crystalline state and became amorphous.

Ebert and Gottlieb (35) showed that Nujol is good for studies of polymorphism since the various crystal modifications show characteristic differences in the infrared. This, however, becomes a disadvantage when studying the spectral changes due to substituents in elucidating structure. Here KBr has the same advantages and disadvantages as Nujol.

Spectral anomalies due to polymorphism occur very frequently in the —C=O region where a splitting of the band may occur, although the effect is often more marked in Nujol than in KBr. For example, the KBr and Nujol spectra of Valamin (1-ethynylcyclohexyl carbamate) show a splitting of the —C=O band at 1724 cm^{-1} and 1681 cm^{-1} , whereas in a chloroform solution there is only a single band at 1724 cm^{-1} (36).

One interesting substance is chloral hydrate which shows different spectra in a mull or a KBr disk. When a Nujol mull of chloral hydrate is run, a medium-strong carbonyl (—C=O) band is observed but the same material run in KBr shows the diol structure predominantly.



A definite shifting of the chemical equilibrium has occurred.

Despite these pitfalls the KBr technique remains popular, with many variations tried by individuals. Some spectrographers use reagent grade KBr (21); others grind old broken cell windows or random pieces of KBr purchased from crystal manufacturers. Nevertheless, for the best work it is advisable to rely on commercially available material having high purity and a particle size between 200 and 400 mesh. In the laboratory it is a good precaution to dry the material at about 110°C , preferably in a vacuum oven. At too high a temperature melting and discoloration may result.

Mixing and Grinding Procedures

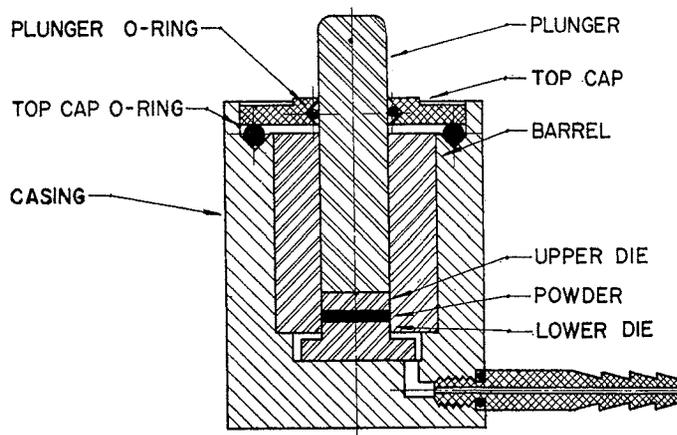
Grinding and mixing methods vary quite widely. The ancient mortar and pestle of agate or Mullite is favored by some but there is always a contamination problem and a great deal of water absorption. Better results may be obtained by grinding in the presence of a volatile solvent (2). Mechanical vibrators have become popular in the last few years, the best ones providing high speed motion along 3 axes to assure rapid thorough grinding and mixing. Hansdorff (22) showed that samples prepared in mechanical grinders gave sharper, more reproducible bands than hand-ground samples.

As an alternate to grinding, Schiedt developed a technique of freeze-drying (2 and 7). This is a lengthy process but produces a very uniform particle size for accurate quantitative work. Weighed amounts of KBr and sample are frozen rapidly in a mixture of dry ice and acetone. The resultant mixture is dried over phosphorus pentoxide at reduced pressure and 50°C to give very small uniform particles, 0.01 to 0.05 microns in diameter. The technique was further extended to microanalysis by Schwartz et al (23).

Pellet Pressing

The sample and matrix, having been ground and mixed, are now ready for pressing in a precision, evacuable die. This must be able to withstand pressures of 25,000 pounds, its moving parts being so well mated that a vacuum of a 1-5 mm Hg can be maintained in the sample in order to remove entrapped air which would cause cloudy pellets. Mating parts should be polished to a mirror surface and be accurately parallel to im-

part these properties to the finished pellet. Clearances between all parts should be so small that no powder is squeezed between them to produce a ragged edge. Choice of material should be such that corrosion does not occur in normal use although it is necessary to clean the parts thoroughly with water and a solvent such as ethyl alcohol.



Before the sample is introduced, the entire die should be warmed to ward off moisture and minimize fogging. With the lower anvil in place in the die body, the mixture is introduced—rolled-up machine-glazed paper makes a convenient funnel. The powder is then leveled by tamping it lightly, a step which cannot be overemphasized. If the powder is high at the edges or uneven over its whole surface, poor pellets are sure to result. After leveling the powder, the plunger is inserted into the die and the entire assembly placed in a hydraulic press capable of providing a ram load of up to 20,000 lbs. pressure held for a period up to 20 minutes, depending mainly on the size of the die. In addition to pellet size, the actual time will depend on the dryness of the powder, the ultimate vacuum and capacity of the vacuum pump.

Releasing the pellet unbroken from the die again poses some problems. Good pellet handling technique is essential. After the pressure is released and the vacuum line disconnected, the pellet is removed from the die by disassembling the latter. When the disk is removed it should be picked up with plastic or rubber gloves or, preferably, transferred directly to the spectrophotometer with tweezers.

Although evacuable dies are to be preferred, plain ones have been used. In these the pellet is difficult to remove and, unless the atmosphere is bone dry, moisture in the entrapped air soon clouds the pellet. An even simpler pelleting method is to eliminate the die entirely and to use instead a hole in a piece of blotting paper. An aperture 1-1/2" by 3/4" is filled with sample KBr and then pressed between two steel disks slightly larger than the sheet of blotting paper at 15,000 psi for 5 seconds. The pressure is released, cloudy spots are sprinkled with KBr and the disk is repressed. By repeating the process several times, a window which is, and will remain, perfectly transparent may be obtained.

Often, even with an evacuable die, a translucent but not transparent disk is produced. Evidently, this depends on the nature of the sample, for satisfactory infrared curves may, nevertheless, be obtained. Some translucent disks are not, however, a result of the sample but of the water-laden atmosphere. If a spectrographer finds the room temperature creeping up in the high 70s and the humidity above 40%, he would do well to use a manometer and a dry ice trap in the vacuum line to reduce the amount of entrapped water vapor. The insidiousness of water vapor cannot be overlooked and precautions must be taken to remove it at every step.

The following table, adapted from a brochure by Research and Industrial Instruments of London, is useful in evaluating when a pure KBr disk is acceptable.

<i>Fault</i>	<i>Cause</i>	<i>Remedy</i>
1. Disk scatters a small percentage of incident light. Good visibility of distant objects through disk.	KBr particle size too large.	Further grinding.
2. Disk shows white spots in a clear matrix.	A few large particles of KBr are still present.	More even grinding.
3. Bad light scatter and poor visibility of distant objects through disk.	Impurities in KBr.	Use purer KBr or purify existing material.
4. "Blotchy" appearance (combination of 1 & 2).	Usually associated with dampness and coagulation of KBr.	Dry and break up powder.
5. Disk flaking.	Excessive grinding of powder.	Grind fresh sample for shorter period.
6. Disk opaque over part of area—often middle.	Insufficient pressure possibly coupled with distribution within the die.	Re-press taking care to level the material within the die.
7. Clear disk on removal from press but development of internal cloudiness on standing.	Lack of vacuum.	Check vacuum and reduce pressure if possible.

Quantitative Analysis

When the KBr method was further extended to quantitative measurements (2, 7, 19, 24) it was soon found that, provided certain precautions were taken, it compared favorably with the liquid-phase method. The technique for preparing the pellet is essentially the same, the sample must be of an optimum particle size so that the Lambert-Beer law is obeyed and the dispersion in the matrix must be uniform.

Lambert's law states that the proportion of light absorbed by a transparent medium is independent of the intensity of the incident light and that each successive unit layer of the medium absorbs an equal fraction of the light passing through it:

$$\ln I_0/I = ab$$

where a = absorption coefficient, b = thickness, I_0 = intensity of incident light and I = intensity of transmitted light.

Beer's law states that the light absorption of a solution is proportional to the number of molecules of absorbing substance through which the light passes. If the above equation is expressed in log to the base 10, then the absorbance A is given by the equation

$$A = \log I_0/I = Kb$$

where K is the extinction coefficient. Hence

$$K = A/b$$

If the molecular extinction coefficient is ϵ and the concentration in gram-mols./liter is c , then

$$A = \epsilon cb.$$

This is the Lambert-Beer law on which most applications of spectrophotometric measurements are based. Although Lambert's law holds for all cases so far investigated, Beer's law cannot be assumed to hold for any particular compound and it is good practice to examine the system for adherence to the law over the range of concentrations to be considered. If pellets of a known concentration of substance in KBr are pressed to differing thicknesses and found to have constant absorptivity, it can then be assumed that pellet thickness is an accurate measure of the concentration (24).

Thus, for accurate measurements, the disks are weighed and their thickness measured at several points on the disk surface with a dial micrometer. Obviously, there should not be a great variation of thickness and the production of uniform pellets of similar weight is essential for quantitative analysis. Weighing techniques depend on the amount of material available. With a large sample, enough may be weighed to within 0.1 mg on an analytical balance before mixing with the KBr. Aliquots may then be taken to form the pellets. If only very small amounts are available, the sample should be weighed on a semi-micro balance or a precision torsion balance, then added to the KBr for direct pellet pressing. Assuming the Lambert-Beer law is obeyed by the system under consideration, a plot of the absorbance of known concentrations of material against per cent concentration will give a straight line. Pellet thickness rather than weight has been shown to be the better measure of material in the beam (19). It is best, when placing pellets in the beam, to have some reference mark on the pellet holder and the instrument so that placement in the beam is always the same. A graph plotted as mentioned above gives a calibration curve from which unknowns may later be read.

The disadvantage of measuring pellet thickness can be overcome by the time-honored use of an internal standard (29). The compound selected as an internal standard should be readily available in a relatively pure form, stable to heat, non-hygroscopic and lastly, it must grind easily and not cause fogging of the pellets. Using an internal standard, it is not necessary to know sample thickness (30): if the sample absorbance is A_0 at wavelength λ_0 , then

$$A_0 = \epsilon_0 bc_0$$

and if the internal standard at λ_1 has an absorbance

$$A_1 = \epsilon_1 bc_1$$

then

$$A_0/A_1 = \epsilon_0 bc_0 / \epsilon_1 bc_1$$

Since ϵ_0 and ϵ_1 are constants at λ_0 and λ_1 and c_1 is made constant then

$$A_0/A_1 = K_2 c_0,$$

where K_2 is a constant. If A_0/A_1 is plotted against c_0 a straight line should be produced. It is not necessary to determine ϵ_0 or ϵ_1 , or even to know c_1 (as long as it is kept constant) to obtain an empirical working curve.

Potassium thiocyanate makes an excellent choice as an internal standard. It should be pre-ground, dried and then re-ground by hand or mechanically, at a convenient concentration, with dry KBr (0.2% by weight of KSCN has been found suitable). If hand-ground, the mixture is quartered, each separately ground, and the quarters re-combined and re-ground. This process is repeated and the final mix stored over phosphorus pentoxide. The reason for this rather laborious preparation is to produce extreme uniformity of the internal standard. A standard calibration curve is then made by mixing about 10% by weight of the material under examination with the KBr-KSCN mixture and then grinding. Different weights of this mixture at convenient increments are mixed with enough KBr-KSCN mixture to give a constant weight pellet. The ratio of the thiocyanate absorption at 2125 cm^{-1} to a chosen band absorption of the sample is plotted against per cent concentration of the sample. Using the same KBr-KSCN mixture, the unknown is formed into a similar pellet, its absorbance ratio measured and the concentration read off the curve. Measurement of sample thickness or proof of obedience to Beer's Law is not required.

Micro Samples

When sample size becomes very small, new techniques of using the KBr method must be devised (31, 32, 33, 34). In many kinds of work, such as in the photographic and plastics industries, inclusions in materials are often so small that it is impossible to collect even 1 mg of material. For these samples, micro-dies have been designed which produce pellets weighing as little as 5 mg. Sample sizes as low as 5 micrograms have been used but generally 50 to 100 micrograms are used to obtain an IR curve without loss of resolution.

A typical micropellet die does not differ basically from its larger brother. It usually forms a pellet from 0.5 mm to 1.5 mm diameter and some produce rectangular pellets (to conform to the shape of the infrared beam) as small as 1 mm by 5 mm. The dies may be of stainless steel, evacuable or unevacuatable, and dies with plastic bodies have been used, too. The most successful die is again the simplest, consisting essentially of three pieces as mentioned earlier. The pellet is now so small that the area of the sample beam becomes very important and to fill the beam of the spectrophotometer, reflecting microscopes have been used. These are, however, expensive and difficult to align. Simple beam condensing systems using AgCl lenses have been designed (31, 32), which effectively reduce the sample beam. The alignment in the reduced beam may be done either by a mechanism similar to the mechanical stage of a microscope or by some system of pins and slots on the sample holder and beam condenser so that constant position is maintained. Several types of beam condenser are available with AgCl or KBr lenses and also all-reflecting optical systems. Kirkland (33) described a die for fractional milligram quantities requiring no pellet holder or beam condensing system. Using his die for both qualitative and quantitative measurements, about 25 to 100 micrograms of sample were required to form pellets 1.8 mm by 13.8 mm. In the instrument used, a Perkin-Elmer Model 21, this is almost the size of the minimum sample beam area. Since the pellet is actually the size of the beam, quantitative application is simplified and the pellets do not have to be too uniform or of reproducible thickness. The controlling factor is homogeneity and small particle size.

Freeze-drying and vibrator grinding were both used in this work.

Conclusions

There is still much argument for and against the mull versus the KBr technique, but there is no gainsaying the fact that, provided precautions are taken, the observance of which is common to all accurate work, the KBr method is extremely useful. By reducing scattering losses and giving a spectrum free of interfering bands it makes accurate, simple quantitative work possible. For ease of preparation and wide applicability the method is hard to beat. That its advantages far outweigh its disadvantages is shown by the extremely wide range of applicability extending from the study of mixed polymers (39) on the one hand to mineral identification, kiln control and quartz analysis on the other (40).

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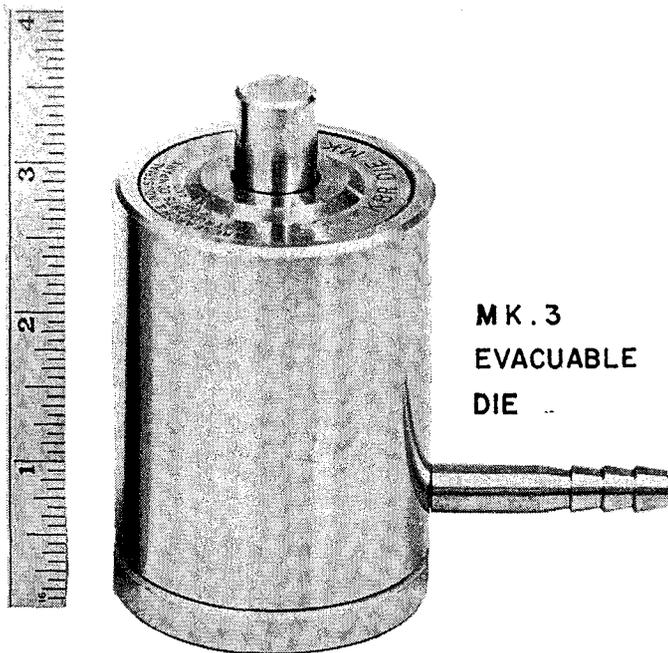
Available in dozens of models and ranges, the Ultramatic is basically a torsion balance in which a precision beryllium copper spiral spring is loaded on one end with the weight to be measured and restored to the unloaded position by turning a large, easily-read dial. The mechanism is suspended—there are no knife edges to wear or damage. Each Ultramatic includes a shallow, rounded pan and an open-end cylindrical trough pan; a weight for checking calibration; horn tipped tweezers; a vinyl dust cover. Easily portable (11 pounds), the balance is corrosion resistant and impervious to external influences such as magnetism, temperature, or humidity. An air dash pot damps out oscillations permitting each reading within 10 to 15 seconds—almost as fast as a single-pan automatic balance three times the cost.

The Ultramatic has, however, a limited range tied in with readability or accuracy. A model with a maximum capacity of 1 mg can be read to an accuracy of 0.001 mg while a 1 gram model can be read to but 1 mg. Thus a correctly chosen unit, while not a substitute for a general purpose analytical balance, is ideal for repetitive weighings.

There is just such an application in the KBr technique. Here about 0.5 mg of sample is mixed with 300 mg of KBr. For qualitative and semi-quantitative work, the Ultramatic recommended is one having a range of 100 mg fitted with a range extender permitting a total range of 500 mg. The readability of this model is such that as little as 0.5 mg of sample may be weighed out, yet the same balance may be used for weighing the bulky alkali halide material. For quantitative work where a weighing accuracy of greater than $\pm 20\%$ is required, the 10 mg model (no range extender is available on balances under 100 mg) is recommended in addition. With it, the sample may be weighed out to an accuracy of $\pm 2\%$.

7002 Sauter Ultramatic Precision Balance, 100 mg
model with range extender permitting weighings up to 500 mg, readability 0.1 mg. **\$275.00**

7003 Sauter Ultramatic Precision Balance, 10 mg
model, readability 0.01 mg. **\$260.00**



**MK.3
EVACUABLE
DIE**

KBr DIE

With well over one thousand in use, the RII KBr die is certainly the most popular for preparing pressed disks. To produce highly transparent disks, the hardened faces between which the material is pressed are highly polished. Precision machining assures perfectly parallel faces and the design is such that disks can be prepared rapidly. Choice of materials—stainless steel and heavily chromium plated tool steel—deter corrosion and make for a long life. Low cost paper rings inside which the powder may be pressed facilitate extraction and handling of disks. Similarly for the preparation of micro-disks, small rectangular paper frames are employed. The die is approximately 3.5 inches in height and 2 inches in diameter.

MK.3 Potassium Bromide Evacuable Die, specify
13, 14 or 15 mm. dia. **\$120.00**

Paper rings for above, specify 13, 14 or 15 mm
dia. **25 \$ 0.80**

Paper Microframes for above, specify instru-
ment **25 \$ 0.80**

POTASSIUM BROMIDE

6004 Potassium Bromide, infrared grade, 200-400
mesh; vacuum dried **100 g. \$10.00**

OUR SALT IS GREEN . . .

And our face is red. What we've been calling copper fluoride under Cat. No. 6003 is apparently copper hydroxy fluoride, CuOHF. One consolation is that it is regularly used in so many laboratories, dropping the detection limit for silicon and boron down to the ppb range. Try it at \$6.00 per 10 g, enough for hundreds of analyses.

CONTROLLING GAS FLOW

Users of the Stallwood jet and inert gas chambers will be interested in a new assembly for control of the gas flow. Consisting of a pressure reducer, regulator, flow meter, toggle valve, hose connection, and adapters to fit most gas cylinders, the unit permits the operator to switch the gas flow off and then back on to a pre-set value. Once adjusted the gas flow rate is thus reproduced from sample to sample.

9024 Regulator-Flow Meter Assembly as described. . \$41.00

Inorganic Cards

Complying with the requests of numerous subscribers, DMS is now adding inorganic spectra to its index. Vol. 4, No. 4 (1960) has 32 such spectra and the number in future issues will depend on the availability and demand.

A special code developed by Dr. H. J. Becher of the Institute of Inorganic Chemistry, Stuttgart, and tested in a number of other European laboratories is used for the inorganic compounds. Cards are identical in shape and size with the organic cards (although colored blue rather than pink) and can be stored and sorted in the same way. The code itself is supplied with the first issue.

Group Spectral Cards (G-Cards)

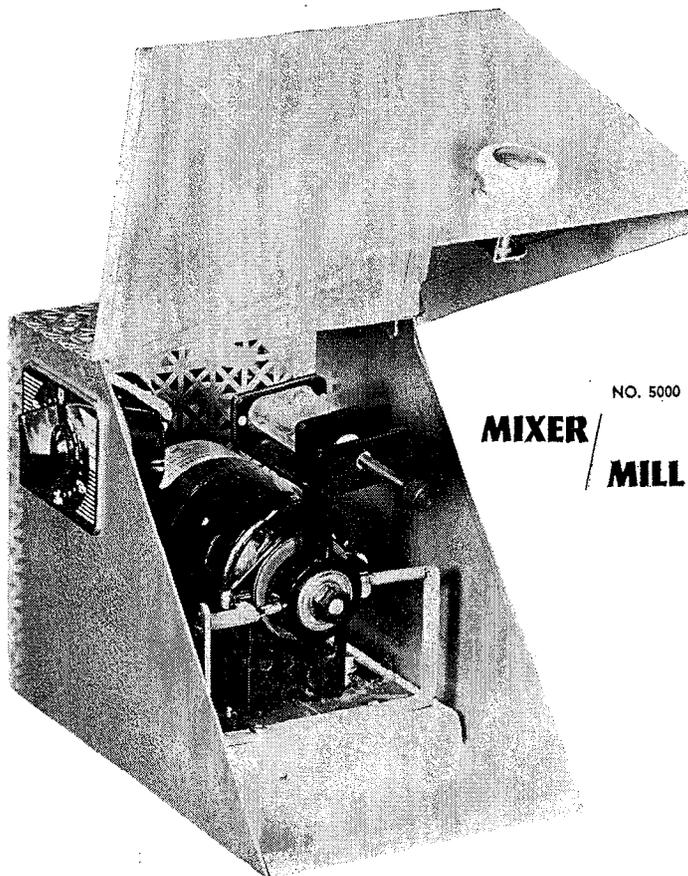
In order to cover papers from the literature dealing with groups of compounds, a small number of G-Cards will be included in future DMS issues, since the L-Card code does not lend itself to the coding of spectral and chemical correlations. The new cards will be on the same pink format but hole 62 will be slotted to indicate G-Card. Only such papers will be included as comprise new and useful contributions to group-frequency correlations.

DMS subscription, Vol. V (issues 17-20)

unslotted \$182.00

slotted \$217.00

Formula List, Vol. IV-VI cumulative\$ 6.00



NO. 5000 MIXER/MILL

Since the discovery of the KBr technique, a number of mechanical grinders have been built for rapidly pulverizing and mixing the sample and alkali halide. The minute sample must be intimately mixed with around 600 times its weight of KBr without loss of sample and with a minimum of contamination from other samples or water vapor. The No. 5000 Mixer/Mill, a brand new instrument, was designed to overcome the shortcomings of older instruments:

- 1) Extremely rugged, a prototype was run at maximum load for several hundred hours without sign of breakdown or wear.
- 2) It has continuously adjustable jaws capable of holding containers up to 2" long and 3/4" o.d.—no need for special adapters.
- 3) A unique timer-switch runs the instrument for periods up to 6 minutes or, at another setting, up to 1 hour.
- 4) To assure rapid grinding, the oscillating speed is upwards of 3200 rpm at full load.
- 5) A safety cover encloses the vial during operation of the instrument.

Like our very popular but larger No. 8000 Mixer/Mill, the high grinding and mixing efficiency of the No. 5000 is a result of its three-dimensional action. Along the axis of the vial there is a component 1" long; at right angles 3/16" wide and up and down the oscillation is about 1/4". The resulting action is consistent with the general shape and size of the vials. That is, at every stroke there is an impact at the end of the vial—over 100 per second—to crush the material rapidly and reproducibly.

The material itself is placed in a small vial made of stainless steel or agate. Where large numbers of samples are run, it is usually advisable to use the former because they are inexpensive. Agate, on the other hand, is by far the best material known for grinding KBr. It is extremely hard, making it possible to polish it highly, and it is chemically inert to almost every known material. Especially when micro samples are used, the high polish on the agate is important in that it cannot trap tiny particles.

- 5000 Mixer/Mill, 115 v 60 cy, or 230 v 50 cy.
(specify)Each \$140.00
- 3114 Stainless Steel Vial, 1/2" dia. x 1" long, 1 ml grinding capacity, including stainless steel ball 1/4" dia.Each \$7.50
- 3118 Agate Vial, 9/16" dia. x 1-5/16" long, 1 ml grinding capacity, including agate ball 1/4" dia.Each \$40.00
- 3127 Hardened Alloy Steel Vial, 3/4" dia. x 2" long, 3 ml grinding capacity, including stainless steel ball 1/4" dia.Each \$14.00

For Mixing Only

- 6133 Polystyrene Vial, 3/4" dia. x 2" long, 10 ml capacity100 \$6.50
.....1000 \$53.00

(Other plastic vials and balls are listed in "Sample Preparation" catalog, available on request.)

For Ashing

- 3120 High Purity Graphite Vial, 1/2" dia. x 1" long, for ashing up to 500°C and grinding without transfer50 \$24.00

30-TON HYDRAULIC PRESS

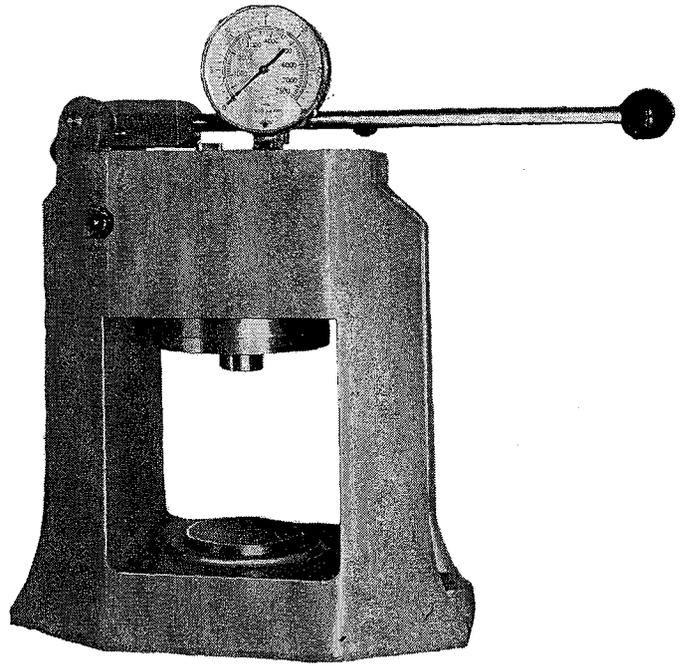
The Research & Industrial Instruments hydraulic press has been designed for pressing a wide range of pellets, tablets or discs used throughout industry. With a total capacity of 67,000 pounds, the press uses but a fraction of its strength for preparing 1/2" dia. KBr discs. Its total capacity is also sufficient to apply a pressure of 40,000 p.s.i. on a 1-1/4" diameter die—the maximum suggested for preparing X-ray pellets. (See "Some Practical Aspects of X-ray Spectrography", M. C. Lambert, *The Norelco Reporter*, Vol. VI, No. 2, 1959, p. 47.)

Built as a virtually maintenance-free machine, the press is cast in one piece from high-strength, non-deforming Meehanite. Sanded smooth, the casting is finished in grey hammertone stoving enamel while all the exposed machine parts have a polished chrome-plate surface or are in stainless steel. A component vulnerable in many presses is the high-pressure oil seal which, in this instrument, actually consists of six high-duty gaskets, one behind the next. The failure of all of them would be necessary for an oil leak to occur.

When used for repetitive work, the die is centered on the stationary platen with the help of a number of concentric rings. The main valve is then closed by turning a handle through 180° and the pressure brought up by pumping the long handle. When the required pressure is reached, the valve is opened momentarily causing the upper moving platen to spring back just far enough to remove the die. This reduces the pumping time for subsequent samples.

Specifications

Pressure	Maximum—30 tons (67,200 pounds)
Platen Stroke	Maximum—1 inch
Vertical Opening	Maximum—5-3/8 inches



Dimensions	15" wide x 9" deep x 16" high
Weight	Net—210 pounds Crated—265 pounds

RH30-1	30-ton Hydraulic Press as described, complete with hydraulic oil	\$550.00
3621	1/2" diameter die, non-evacuatable	138.00
3622	1-1/4" diameter die, non-evacuatable	210.00

SPEX

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