May 22, 1962 R. N. JONES ET AL 3,036,215

SPECTROMETRIC CELL STRUCTURE

Filed April 30, 1959 3 Sheets-Sheet 1

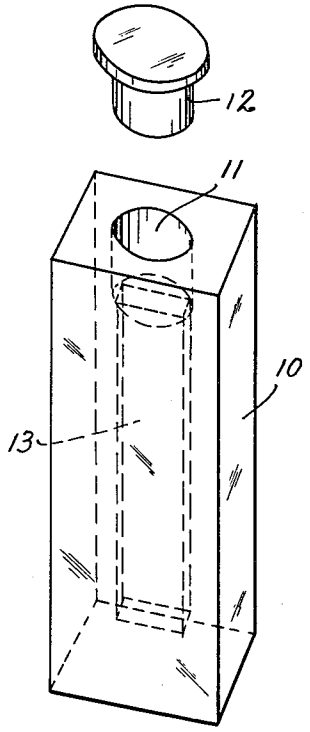


Fig. 1.

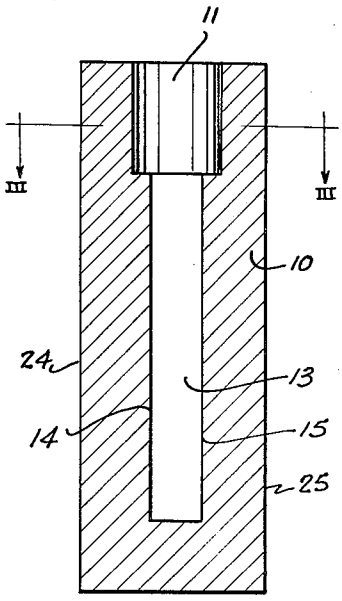


Fig. 2.

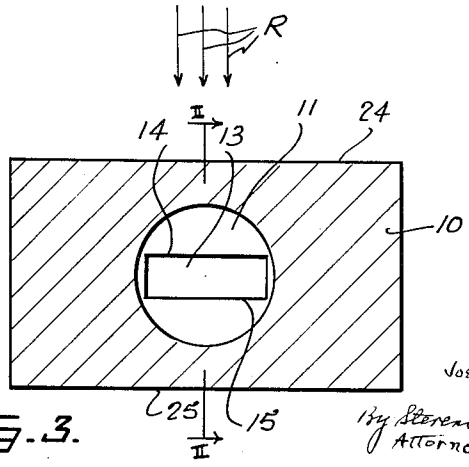


Fig. 3.

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SPECTROMETRIC CELL STRUCTURE

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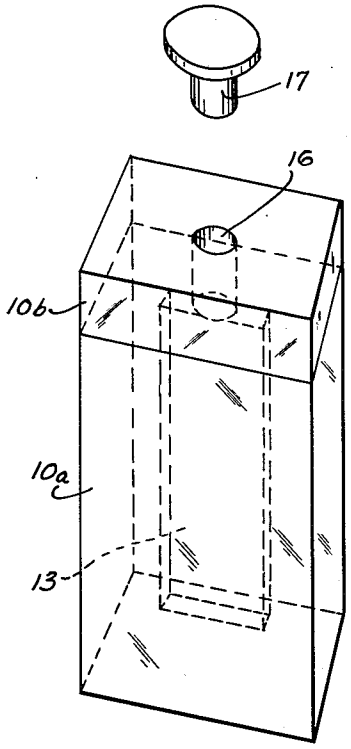


Fig. 4.

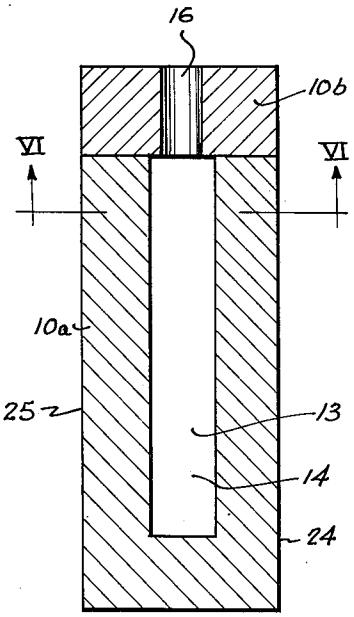


Fig. 5.

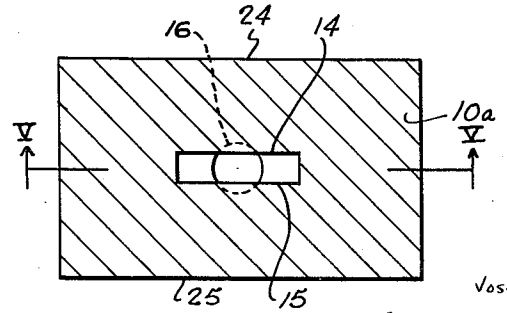


Fig. 6.

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SPECTROMETRIC CELL STRUCTURE

Filed April 30, 1959 3 Sheets-Sheet 3

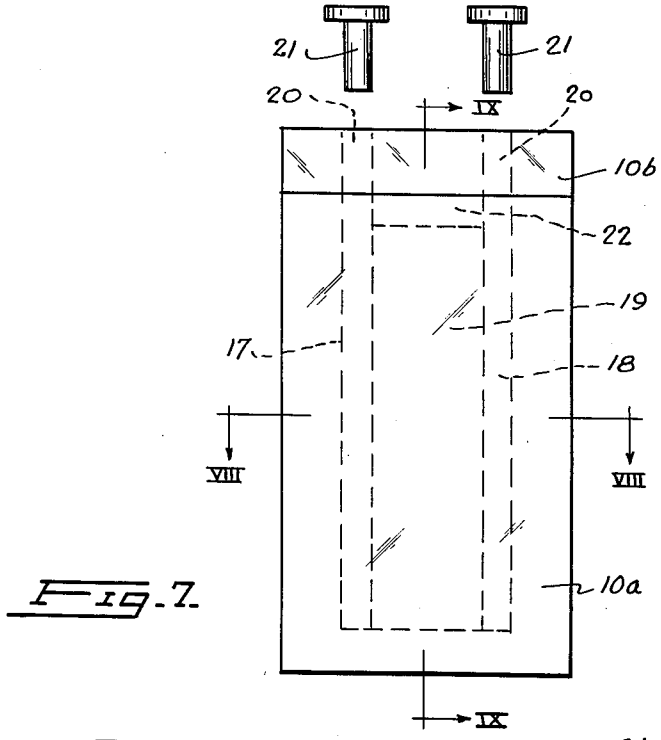


Fig. 7.

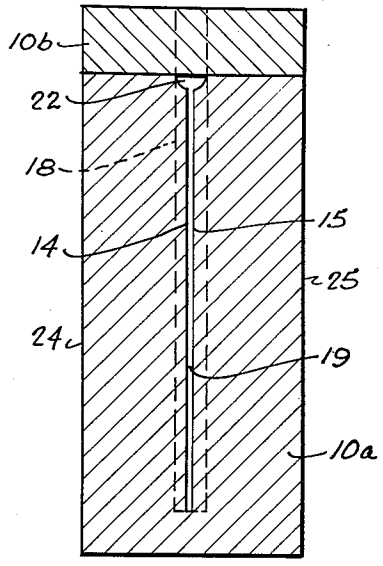


Fig. 9.

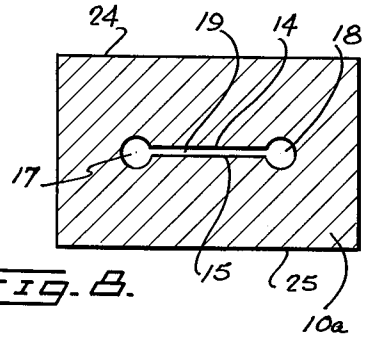


Fig. 8.

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3,036,215

SPECTROMETRIC CELL STRUCTURE

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4 Claims. (Cl. 250—43.5)

This invention relates to the construction of cells of the type employed for mounting specimens for spectrometric examination. The use of spectral analysis, particularly infra-red spectral analysis, is now a well-established laboratory technique for the identification of organic compounds, and infra-red spectrometers have become standard equipment in most laboratories concerned with organic chemical analysis.

The preferred method at present in use for mounting a sample in a spectrometer is to dissolve the sample in a suitable solvent, and then introduce resulting specimen into a cell-containing device which is mounted in the spectrometer. The term "sample" is used herein to denote the material for analysis; the term "specimen" is used to denote the combination of sample and vehicle that is placed in the cell for examination in the spectrometer. The basic requirements for the solvent that is used as the vehicle are that it should be chemically inert both towards the sample and towards the material of which the cell-containing device is constructed. Also, of course, the sample must be sufficiently soluble in the solvent. Moreover, it is necessary that the solvent have satisfactory optical properties, that is, it must be optically transparent to the radiation being employed in the spectrometer. To be optically transparent the solvent must not only transmit the radiation employed in the spectrometer, but it must not modify such radiation to any appreciable extent, since any such modification would swamp the characteristic spectral modifications imparted to the radiation by the sample in solution.

The present invention is concerned with improvements in the construction of devices which contain a cell in which such a specimen can be mounted in a spectrometer for spectral analysis. Cell-containing devices in use at the present time are of comparatively complex structure usually involving many separate pieces of different materials, often including precision made metal parts fabricated into a complex assembly. As a result, these devices are expensive, and it is only economical for a laboratory to keep on hand a comparatively small number of them. They must be thoroughly washed out after each use and carefully stored for further use. This economic need for the frequent reuse of known cell-containing devices is also disadvantageous in that it reduces the utilization of the spectrometer, since this instrument will normally have to remain idle while each cell-containing device or a group of such devices is washed out and recharged with a new specimen.

A principal object of the present invention is to provide an improved cell-containing device that will be considerably simpler than existing devices, and consequently cheaper to manufacture, while still performing satisfactorily in the spectrometer. By cheapening the manufacture of such devices, it becomes practicable for a laboratory to have on hand sufficient of the devices for a large number of them to be charged in advance and fed into the spectrometer without delay, thus obtaining maximum utilization of this expensive instrument and of the time of the skilled technician who operates it.

If an infra-red examination is to be made, the cell-containing device will be made of a material optically transparent to infra-red radiation. Numerous suitable

materials are known. These are normally chosen from the alkali metal halides, although a silver halide is sometimes used. Other materials that may be used are barium, calcium and lead halides; also arsenic trisulphide. Sodium chloride in the form of natural or synthetic rock salt is the most readily available material commercially. When rock salt is used, there is a requirement that it must be in the form of a single crystal, but there is no difficulty in obtaining crystals of rock salt of sufficient size, since they can be readily synthesized. Typical dimensions for a cell-containing device of this type would be about two centimeters long, one centimeter wide and 0.5 centimeter thick. Pieces of rock salt crystal of this comparatively small size are readily available as scrap left over from the manufacture of larger optical bodies.

If ultra-violet or visible radiation is to be employed in the spectrometer, the material of the carrier body may be quartz crystal, or in some cases one of the optical glasses available and having suitable optical transparency.

It will be assumed, however, for the purposes of the subsequent specific description, that infra-red spectrometry is concerned and that the cell-containing device is formed from rock salt, since, although the invention is essentially sufficiently broad in scope to include the other forms of radiation and other materials indicated above, infra-red spectrometry is by far the most common form of spectrometry employed and rock salt is the most convenient material to employ.

In our previous United States Patent 2,927,209, issued March 1, 1960, there is disclosed a device containing a cell which may be of a size suitable for containing a "macroscopic" sample (about 1 milligram); a "microscopic" sample (about 100 micrograms) or an "ultramicroscopic" sample (50 micrograms or below).

The present invention is concerned with cell structures designed for use with specimens containing samples in the macroscopic and microscopic ranges, but not in the ultramicroscopic range. In the ultra-microscopic range, the specimen is so small that the shape of the cell is of little or no importance. With the larger specimens involved with microscopic and macroscopic samples, however, the shape of the cell becomes important. In particular, it becomes necessary for satisfactory optical performance to provide the cell with a pair of planar side walls, parallel to one another, through which the radiation enters and leaves the cell. It is principally this requirement that has led to the comparatively complex construction previously employed in most cell-containing devices. With few exceptions, devices in current use consist essentially of a pair of flat plates of optically transparent material separated by a spacer. Access to the cell cavity is provided by holes drilled through the edge of the spacer, or through the face of one of the windows. Unless carefully constructed and maintained, such devices are subject to leakage. If the windows and spacers are sealed with a chemical adhesive, leaks may develop through attack on the adhesive by the solvent, and products extracted from the adhesive may contaminate the sample and invalidate the spectrum. In cell-containing devices of more recent design, these difficulties have been overcome by careful polishing and machining of the component parts so that a leak resistant seal between cell window and spacer can be achieved by the application of pressure alone. Such cells, however, are expensive to produce, and their servicing and replacement is a significant economic factor on the maintenance of an infra-red spectrometer in routine operation.

In contrast to such complex structures, the present invention comprises a unitary body formed from a single piece of material, said body being provided with a cell cavity having a pair of planar faces extending parallel to each other. To form such a device it is necessary to hollow out the material into an elongated cavity of rec-

tangular cross-section or other cross-section having a pair of flat and parallel opposite faces. This can be done by the impact grinding technique described below.

For further details, reference is directed to the accompanying drawings which illustrate three forms of cell-containing device constructed in accordance with the present invention.

FIGURE 1 is an exploded perspective view of a first form of cell-containing device;

FIGURE 2 is a central vertical section of the main portion of the device seen in FGURE 1 taken on line II—II in FIGURE 3;

FIGURE 3 is a transverse section taken on the line III—III in FIGURE 2;

FIGURE 4 is an exploded perspective view of a second cell-containing device according to the invention;

FIGURE 5 is a central vertical section of the main portion of the device seen in FIGURE 4 taken on line V—V in FIGURE 6;

FIGURE 6 is a transverse section taken on the line VI—VI in FIGURE 5;

FIGURE 7 is a front elevation view of a third form of cell-containing device constructed in accordance with the present invention;

FIGURE 8 is a transverse section taken on the line VIII—VIII in FIGURE 7; and

FIGURE 9 is a vertical central section taken on the line IX—IX in FIGURE 7.

The device seen in FIGURES 1–3 consists of an elongated body **10** of a single crystal of rock salt drilled centrally to provide a first cavity **11**, which will be referred to as the "neck." This neck **11** is of circular cross-section and serves to receive a stopper **12** (shown only in FIGURE 1) formed of a resilient material inert both to to solvent and to the rock salt. A suitable material is the synthetic resin known under the trademark Teflon. Inwardly of the neck **11** and communicating therewith is a second cavity **13** which constitutes the cell itself. This cell **13** is rectangular in cross-section so as to provide a pair of parallel planar faces **14** and **15** (FIGURE 3). Whereas the neck **11** is formed by conventional drilling, the cell **13** is formed by ultrasonic impact grinding. The general technique for producing small cavities in brittle materials by ultrasonic impact grinding has been described by M. S. Hartley in Electronics, January 1956, pages 132–135. A bit of approximately the cross-section of the cavity to be made is pressed down against the material while being oscillated at an ultrasonic frequency of 25 kilocycles. Initially a cavity is produced using a slightly undersized mild steel bit fed with No. 218 mesh Carborundum abrasive suspended in absolute ethanol. The walls of the cavity so produced have a coarsely etched appearance. A preliminary polish is obtained by repeating the drilling process with a slightly larger bit and a fine emery powder. A final smooth surface is achieved by flushing out the cell rapidly with 50% aqueous ethanol followed immediately by absolute ethanol.

For best optical properties, the outer faces **24** and **25** of the body **10** will be ground and polished parallel to one another and the faces **24** and **25** will be parallel to faces **14** and **15**. Some deviation in the planarity and parallelism of faces **14** and **15** can be tolerated. In a spectrometer the radiation will pass through the device as demonstrated by the arrows R.

A modified form of cell is illustrated in FIGURES 4–6. In this case the body of the cell-containing device is formed in two parts, a lower main, specimen-mounting part **10***a* and an upper part **10***b*. Before parts **10***a* and **10***b* are joined together, a rectangular cell cavity **13** is formed in the lower body part **10***a* by impact grinding in the same manner as just described. Parallel planar faces **14** and **15** are thus provided as before. Independently of this operation a neck hole **16** is formed in the upper body part **10***b*. This has to be done separately as the diameter of the neck **16** is smaller than the width (the larger cross-sectional dimension) of the rectangular cell **13**. When these two cavities have been made, the two parts **10***a* and **10***b* of the body are brought together and bonded. To effect this bonding the contact surfaces of the two parts of the body are smoothed, moistened with saturated aqueous sodium chloride solution, placed together and then clamped under pressure with heating to 110° C. for twelve hours. Strong bonds between two bodies of rock salt can be obtained in this manner and the bond will resist cleavage at temperatures up to 200° C. The bonded surface is not optically clear, but this is no disadvantage, since this surface is not placed in the optical beam in the spectrometer. Stopper **17** is provided to close neck **16**. This second construction has the advantage over the construction shown in FIGURES 1–3 that the neck **16** is of smaller diameter than the neck **11** and can thus be more effectively sealed. With the wider neck of the first embodiment it is more difficult to avoid solvent evaporation and greater care must be exercised to ensure a tightly fitting stopper while the cell is in use. On the other hand, the first embodiment is cheaper to manufacture as not involving the two-part construction and bonding step.

In both types of cell-containing device so far described the thickness of the cell (i.e. the smaller of the two dimensions in cross-section, the distance between faces **14** and **15**) is approximately one millimeter. This is a substantial distance for the radiation to traverse through the specimens and as a result these cells are satisfactory with comparatively dilute solutions. With solutions of greater opacity, it is desirable to have a shorter optical path through the specimen if good results are to be obtained in the spectrometer. It is possible to form cavities narrower than 1 millimeter in rock salt by the ultrasonic drilling technique, but the bits employed become very thin and delicate and tend to deform during the grinding process producing curved cavities that are no longer parallel with the outside faces of the body. This is undesirable for optical reasons.

This difficulty can be overcome by adopting the shape of cavity illustrated in the third embodiment shown in FIGURES 7–9, where a cavity is formed with a cross-section of dumb-bell shape. A dumb-bell bit is employed comprising a pair of rounded side members of approximately 0.75 millimeter diameter connected by a cross-bar of approximately 0.1 millimeter thickness to form the actual functional slot portion of the cell cavity. Such a bit forms a cell cavity of complex shape having a pair of cylindrical chambers **17** and **18** joined by a slot portion **19** which is very narrow in comparison with the cells **13** of the previous embodiments. Nevertheless such a cavity can be formed with planar parallel faces by the ultrasonic impact grinding technique, because of the rigidity imparted to the bit that forms the cavity by the cylindrical portions of that bit which forms chambers **17** and **18**.

In use the optical beam traverses only the functional slot portion **19**, of the cell cavity, this slot having parallel planar faces **14** and **15** as before. As in the case of the structure shown in FIGURES 4–6, the body of the device shown in FIGURES 7–9 is made in two parts a lower part **10***a* and an upper closure parts **10***b*. The upper part **10***b* is formed with a pair of circular holes **20** each to receive a Teflon stopper **21**. In manufacture the two parts **10***a* and **10***b* are bonded together in a manner similar to that already described, the holes **20** being in register wtih chambers **17** and **18** to enable the cell cavity to be filled and there being no slot in the closure part **10***b* equivalent to the slot **19**. In order to avoid capillary flow of sodium chloride solution down the slot **19** during the bonding process, the upper end of such slot is enlarged into semi-circular cross-section as shown at **22**. When this device is filled with a specimen, the two cylindrical chambers **17** and **18** represent an appreciable parasitic volume, but the capillary action of the very narrow

slot 19 is such that it will remain filled with liquid even when the cylindrical chambers are themselves only partly filled or even empty. In this way the parasitic volume, if it should be disadvantageous, can be almost wholly eliminated by placing only enough specimen in the device to fill the slot 19.

We claim:

1. A device for mounting a liquid specimen for spectrometric examination, at least the specimen-mounting portion of said device comprising a unitary body formed from a single piece of a material chemically inert to the specimen and optically transparent to the radiation of the spectrometer, said body being provided with a cell cavity for containing said specimen, said cell cavity having an elongated chamber and a narrow slot portion, one edge of said slot portion opening into and communicating with said elongated chamber along the length thereof, said slot portion being defined by a pair of planar, mutually parallel faces disposed so close together that liquid specimen will be retained for spectrometric examination in said slot portion by capillary action even when the total volume of said specimen is insufficient to fill said chamber to the same level as said slot, said chamber being too wide to retain liquid specimen therein by capillary action, the dimensions of said slot portion as determined by the extent of said faces in their own planes being substantially greater than capillary size.

2. A device for mounting a liquid specimen for spectrometric examination, at least the specimen-mounting portion of said device comprising a unitary body formed from a single piece of a material chemically inert to the specimen and optically transparent to the radiation of the spectrometer, said body being provided with a cell cavity for containing said specimen, said cell cavity being of dumb-bell shape in cross-section, the central portion of such dumb-bell shape being defined by a pair of planar

faces parallel to each other and disposed so close to each other as to define a narrow slot portion in which said liquid specimen will be retained by capillary action.

3. A device for mounting a liquid specimen for spectrometric examination, at least the specimen-mounting portion of said device comprising a unitary body formed from a single piece of a material chemically inert to the specimen and optically transparent to the radiation of the spectrometer, said body being provided with a cell cavity for containing said specimen, said cavity having a pair of planar faces extending parallel to each other to define a slot portion therebetween, the width of said slot portion as determined by the spacing apart of said faces being of capillary dimension while the length and breadth of said slot portion as determined by the extent of said faces in their own planes both being substantially greater than capillary dimension whereby a sheet of said liquid specimen having only one dimension of capillary size will be retained by capillary action in said slot portion.

4. A device as claimed in claim 3, wherein said slot portion communicates with at least one other portion of said cell cavity, all the dimensions of said other portion being greater than capillary in size whereby such other portion provides a substantial space non-competitive with said slot portion for the volume of liquid specimen required to charge said slot portion but available to contain liquid specimen in excess of said volume.

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