Technique for Filling Cells with Mineral Oil Mulls of Air- and Moisture-Sensitive Substances without Dry Box in Infrared Spectroscopy

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The apparatus and sampling procedure for filling of cells used in infrared spectroscopy in inert atmosphere and without the use of dry box are described.

Infrared spectra of powdered solid substances which undergo changes on contact with air or under the influence of pressure or evacuation, and consequently the technique of pressed discs cannot be used, can usually be obtained after dispersion of the examined substance in the liquid paraffin (Nujol), in perfluorokerosene (Fluorolube), in hexachlorobutadiene, *etc.* The suspension of substances sensitive towards the active components of air (O_2, H_2O, CO_2) has to be protected by the atmosphere of inert gas (argon, nitrogen) during their preparation and filling in cells for infrared spectra measurements.

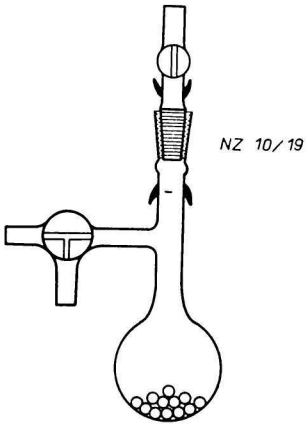
For this purpose indecomposable cells of alkali halides used to be taken. These, though very suitable for filling with liquids, are not suitable for filling with viscous mulls. A conveniently adjusted plate assembly is usually filled with the aid of a syringe in a glove box or some other measures are taken to prevent the access of the air [1—3].

In the present paper a device and a procedure are described which, for the purposes of the infrared spectroscopy, were found to be satisfactory for mulling of air-sensitive highly reactive powdered solid substances and for filling decomposable plate assembly of alkali halide or polyethylene discs, *etc.* with mulls of these substances without the use of a glove box.

Apparatus and procedure

The mull may be prepared under inert conditions by agitating the powdered specimen with the mulling agent (mineral oil), together with glass or steel beads in a sufficiently thick-walled flask (Fig. 1) for several hours. The flask with the beads is first repeatedly evacuated and purged with inert gas. Then the gas is brought into the flask through a T-bore tap; it gets out through a straight-bore tap. This tap is also used for charging the flask with the appropriate amount of mineral oil, *e.g.* with the aid of a hypodermic needle. When the gas flow has been sufficiently raised, the flask neck is opened and through it the powdered sample from the glass ampoule, which has been opened immediately before, is put into the flask. The flask is then plugged, both glass taps are closed and the flask contents are ground and homogenized by agitation for several hours. The mull may be taken from the flask either by overpressure or with the aid of a hypodermic needle through the straight-bore tap when simultaneously nitrogen is being introduced through the T-bore tap.

Fig. 1. Flask for preparation of the mull.



Thus obtained specimen is then transferred between the alkali halide plates mounted in the apparatus wherein provisions have been made for keeping inert atmosphere.

The apparatus consists of a glass tube (Fig. 2) closed by metal attachment wherein the alkali halide plates are held (Fig. 3). This device consists of a metal (duralumin)

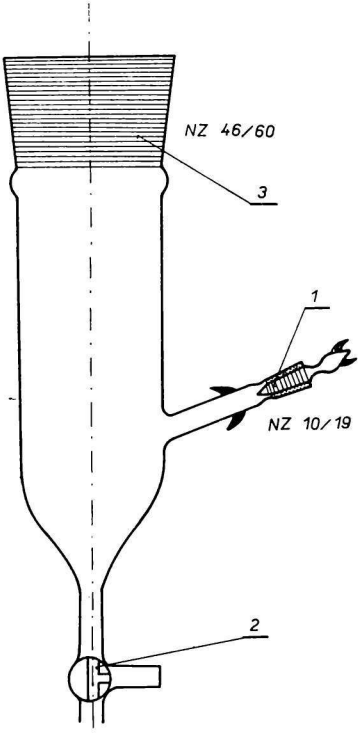


Fig. 2. Glass tube.

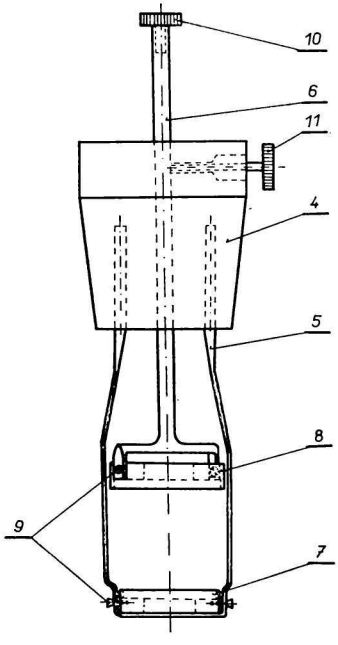


Fig. 3. Metal attachment.

ground stopper *4*, of a fixed support for the lower disc holder *5* and of a movable metal support of the upper disc holder *6*. The disc holders *7*, *8* made of an appropriate material (duralumin, teflon) are connected with the supports with the screws *9*. The movable support *6*, terminated by a manipulation piece *10* passes through a tight opening in the middle of a stopper *4*, where it may be fixed in the chosen position by the screw *11*.

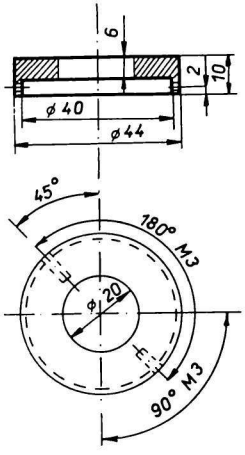


Fig. 4. Holder.

Before the whole device is placed in the glass tube, the alkali halide plates are fixed in the holders *7* and *8* (see also Fig. 4), *e.g.* with the aid of screws. The lower holder remains in the horizontal position, the upper holder is placed at a sloping position of about 45° and is fastened by the screw *11* so that it nearly touches the lower holder. Rotating the stopper in the neck of the glass flask *3* the holders are placed so that the part of the upper holder *8* is opposite to the glass tube *1* closed by a stopper.

Before a drop of the mulled specimen is introduced through the tube *1* to the disc mounted on the lower support *7*, the whole apparatus is thoroughly purged by inert gas. In the course of the whole procedure the apparatus is protected against the infiltration of the air by a slight overpressure of the inert gas introduced through the T-bore tap *2* which is allowed to get out through the tube *1* along the support *6*.

The mull drop in the middle of the disc *7* is, after the screw *11* has been loosened, covered by the upper disc *8* which, under the action of a slow pressure, gets from vertical to horizontal position. A uniform and sufficiently thin film of mineral oil mull may be conveniently obtained by a rotatory manipulation, brought about by the rotation of the piece *10*, of the upper disc pressed against the lower disc. The two discs pressed against each other are then fixed with the aid of the screw *11* and through the tube *1* a suitable cementing agent (*e.g.* acetone lacquer) is introduced in order to make these discs air-tight also after they have been removed from the apparatus. When it is convenient, the sealing may be applied also to the edges of the horizontal part of the lower disc by which better sealing of both discs may be achieved.

After hardening of the cement the whole piece is removed from the tube and the sealed discs are placed in the standard sample beam path of the infrared spectrophotometer.

Easy and rapid handling is an advantage of this method for which neither a dry box nor any changes in construction of the sealed cells are required. Since standard alkali halide plates, polyethylene plates, *etc.* are sealed it is possible to prepare a stock of any amount of specimens for measurements. The sealed discs can be taken apart by submerging them in a suitable soluting agent (*e.g.* acetone).

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