A handbook of chemical manipulation

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Section XIV. Disintegration, &c.

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DISINTEGRATION, &c.

248. It has already been observed that the attraction of aggregation is one of the greatest obstacles to chemical action, and that the further the particles of bodies are separated from each other, the more freely and easily are they attacked by chemical reagents; it will be seen, therefore, that any means enabling the chemist to effect the disintegration of bodies, should be thoroughly familiar to him.

There are many methods by which solid bodies may be reduced to powder, but only a limited number of them are capable of being used in researches; this arises from the fact that there are but few processes possessing the necessary but somewhat incompatible qualifications of not only pulverizing substances, but at the same time preserving their purity. The ordinary operations of grinding by means of stones, slabs and mullers, mills, &c., will not be noticed in this work, as they are almost solely applicable to manufacturing and technical pursuits; it is true that there are occasions on which the scientific chemist is obliged to avail himself of their assistance, but such are of comparatively rare occurrence.

249. The manner of proceeding to reduce a substance to a fine state of division necessarily depends upon its physical characters; for example, a piece of chalk and an ingot of silver require very different kinds of procedure, the first being easily brought to almost any degree of fineness by mere trituration, the second being only procurable in a pulverulent form by precipitation or filing.

250. The instruments most commonly used by the chemist for the purpose are the pestle and mortar, and these are necessarily made of many different forms and varieties of material, to suit the particular cases which present themselves. Porcelain, glass, agate, porphyry, marble, granite, iron, steel, brass and bell-metal all being in use for various purposes, but only four of them are

employed in the laboratory, namely, porcelain, agate, steel, and iron. Under the head of porcelain is of course included the extremely valuable Wedgwood-ware mortars so much in use. It is considered unnecessary to enter into a description of the best shape adapted for a mortar, because those last alluded to are generally, perhaps always, made upon an unexceptionable model, and the other varieties of form in use in the laboratory are all appropriate in certain processes. In the operation of pulverization, as in most others, much time and trouble may be saved by a systematic method of procedure, and an equally great loss incurred by careless manipulation. Some substances, such for instance as sugar, when touched by the pestle, fall immediately into small fragments, and are easily reduced into fine powder by a grinding motion; others, like sulphuret of iron, require a great deal of pounding, and some care is necessary to prevent dispersion of the fragments; this may be prevented by striking with the pestle straight down, carefully avoiding lateral blows, it being these which chiefly cause the particles to fly about. After the substance has become tolerably reduced, it is better to substitute a movement between that of a blow and a grind for the directly downward motion of the pestle; this is effected by grasping the handle firmly in the hand, the striking portion of the pestle pointing somewhat inwards, and then striking downwards, drawing the pestle towards the operator's body; when by this means a certain degree of fineness has been obtained, it is advisable to entirely substitute trituration for blows, the pestle travelling in turn over the whole of the lower portion of the inside of the mortar. Where the matter adheres to the inside, it must be scraped down towards the centre with a spatula. It is necessary to have only a moderate quantity in the mortar at once, as, otherwise, the fragments are protected by each other from the action of the pestle. If the contact of organic matter is unimportant, it is convenient, and assists the operation, to sift the matter as fast as it is pounded, and return the fragments which are too large to pass through the sieve into the mortar to be repulverized.

A good method of sifting small quantities of matter where sieves would be inapplicable, is to put the powder into a piece of muslin of the proper fineness, and gathering up the ends, to strike the end of the pestle gently with the bag thus formed, over a sheet of paper, of course avoiding a draught, if the powder, from its small density, is liable to be blown away.

251. It is always best to have the pestle formed out of one piece of porcelain, instead of the old plan of cementing a wooden handle into a Wedgwood foot. If the pestle is used for pulverizing oxide of copper or chromate of lead this is indispensable, as it is sometimes difficult to prevent fragments of the cement being projected into the mortar.

252. It is important to ascertain the soundness of the material of which the mortars are made before using them, as a bad one is worse than useless, and sometimes gives rise to serious errors. One of the worst faults is porosity; if sulphuric acid be put into a mortar and left for some hours, it ought to be immediately and thoroughly removed by merely rinsing with cold water; if, moreover, any colouring matter be left in it for twenty-four hours, the same ought in general to be the case. Some unglazed mortars, however, although in other respects excellent, have a tendency to slightly retain colouring matters with great obstinacy, while acids or other fluids do not appear to be absorbed. It is essential that the mortars should be sufficiently hard to allow of substances of considerable hardness being pulverized without injury. It is sometimes found that the pestles and mortars, although purchased together, are not of the same degree of hardness, very hard mortars being sometimes accompanied by worthless pestles, and *vice versâ*; where this is the case, it is better to throw away the imperfect instrument than to run the risk of causing the failure of an experiment by retaining it in the laboratory.

253. When a good instrument has been procured, every care ought to be taken to preserve it. Sudden changes of temperature are to be avoided; many mortars have been destroyed through careless persons mixing sulphuric acid and water in them, or plunging them into boiling water for the purpose of cleansing.

Impurities may be removed from mortars by rubbing in them a little sand saturated with a strong acid or alkali, as the case may be; it is seldom that any dirt is found to resist the action of nitric or sulphuric acids or caustic potash. With the same view, namely, to prevent destruction of a good mortar, care should be taken to prevent unnecessarily hard blows being used in pulverizing; moreover, where it is essential to warm the mortar, as is the case in the comminution of very deliquescent substances, it ought to be done very gradually (such as by letting it remain for some hours in the warm closet) before attempting to make it hot.

254. The operator cannot be too strongly cautioned against pulverizing a chemical mixture with the properties of which he is unacquainted; this especially applies to anything in which chlorate of potash enters as an ingredient, several fearful accidents having occurred in this manner.

255. It has been said that some kinds of unglazed mortars retain colouring matters with obstinacy; it must not be inferred therefore that glazed mortars are the best; on the contrary, and even where the glaze exists, it is only for a short time, as the attrition from use soon removes it, and, in fact, a glaze is an obstacle to the use of a mortar, its polish enabling the substance to slide over its surface without meeting with the proper degree of attrition.

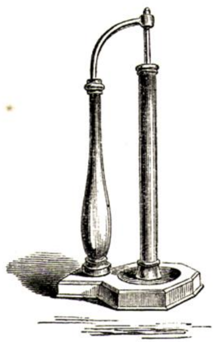
256. It often happens, especially in the preparation of refractory silicates for analysis, that substances are to be reduced to an extreme state of division; it is then generally best to commence by reducing the mineral to coarse fragments, by first heating it to redness and then quenching it in water; this fills the mineral with innumerable fissures, which greatly assist the further progress of the disintegration. It must not however be assumed that this is invariably a correct mode of operating, because there are instances where such a procedure would destroy the possibility of obtaining an accurate result.

257. A very good general method of reducing hard substances into moderate-sized fragments, either after heating to redness and

then quenching or otherwise, is to wrap the mass in a few folds of paper, and holding it with the forefinger and thumb of one hand on the anvil, to give it a few smart blows with a hammer more or less heavy, according to the hardness of the substance. Some bodies, as the phosphuret of rhodium, are very difficult to reduce to fragments even by this method, and are perhaps best broken in the diamond mortar to be described presently. After breaking the mineral to moderate-sized fragments, it may generally be reduced to a coarse powder in an ordinary Wedgwood or Meissen porcelain mortar, and be finished by long trituration in the one of agate.

258. These last-named instruments are absolutely indispensable in the laboratory. The demand for them having greatly increased during the last few years, they may be procured at very low prices, and are therefore within the reach of almost all who study the science to any extent. They are only available for trituration, having generally more or less fissures, which weaken them so much as to cause them to fly to pieces with a smart blow. They are, however, excessively hard, and consisting almost entirely of silica, the nature of any matter introduced may be known and estimated accordingly. The pestles are generally inconveniently short, and therefore should fit into a handle, or the whole arrangement may be made as in fig. 142, where the mortar is fixed and the handle is attached to a tube, which, from the mode of supporting it, allows of a considerable amount of motion, and at the same time greatly facilitates the working of the instrument. It will be seen that the agate mortar from its shape is also singularly well adapted for the pulverization of small quantities of valuable substances for analysis, and should therefore find a place among the apparatus for organic as well as inorganic analysis; it is also cleaned with extreme facility, partly

Fig. 142.



in consequence of its shape, and partly from the perfect absence of porosity, that is at least in good specimens, for some agate mortars contain flaws upon the basin; these should be rejected. In examining them they should be held against the light, when many imperfections which would otherwise pass unnoticed will become visible; care must, however, be taken not to mistake the lines and streaks (almost always to be found in agate) for flaws.

259. One of the great uses of an agate mortar is in qualitative blowpipe analyses, for procuring the metallic globules formed by exposure of oxides with carbonate of soda, &c. to the reducing flame; the charcoal, after the blowpipe process is ended, is cut out at the spot where the action took place, and is ground in the agate mortar, after which the powder is carefully washed away, and the grinding and washing continued, sometimes alternately and sometimes together, until nothing remains in the mortar but the reduced metallic globules, which may generally be recognized by their physical characters, or, if not, by a few simple tests. The reason why only an agate mortar is properly applicable in this process, is, because the rough surface of an ordinary mortar would almost infallibly destroy the globules. A porcelain capsule and the polished end of a porcelain pestle may sometimes be substituted for the agate, where the latter is not to be procured. Great care must be taken in this process to avoid too powerful a stream of water, which would wash away the globules with the charcoal. Several other precautions are also to be taken at times, which belong to analytical treatises, and cannot therefore be entered upon here.

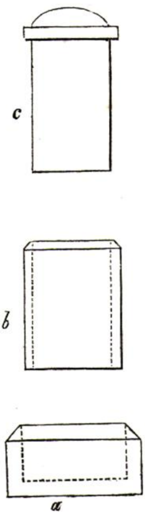
260. An iron mortar is useful for many purposes, and should therefore find a place in the laboratory. It is well adapted for pounding the hard masses of fused chromate of lead and sulphuret of iron, the former used in organic and the latter chiefly in inorganic analysis. It is constantly used, moreover, for pounding marble and several other hard substances.

261. The student should endeavour to familiarize himself with the physical and chemical characters of substances, in order to be able to perform with facility any operations of solution or dis-

integration that may be required, it being well known that different bodies require very different methods of reducing them to powder; for example, camphor is a somewhat troublesome substance to reduce to fine powder *per se*, but if it be moistened with alcohol, it is readily obtained in the required state. Chloride of ammonium is extremely tough if struck at right angles to the grain, but if the blows are given in the direction of the fibres it yields readily.

262. Metallic gold may be obtained in powder by several chemical and mechanical methods: in the first place, it may be precipitated by a solution of protosulphate of iron or by oxalic acid, or it may be ground with honey to a paste, and the honey may afterwards be removed by water. Tin and lead may conveniently be pulverized by melting them and pouring into a wooden box, which is to be vigorously agitated as rapidly as possible after the introduction of the fluid metal.

Fig. 143.



263. Before levigating hard minerals, it is usual to reduce them to a certain degree of fineness in the diamond mortar (fig. 143), consisting of three pieces, *a*, *b*, and *c*; the pestle, *c*, fits into *b* very accurately, so that no particles can be lost by dispersion, and *b* in its turn fits into *a*, which serves as a foot and steadies the whole. When the mineral is inserted into the cavity of *b*, the pestle is put into its place and struck smartly with a hammer, until it is found that the mineral has yielded, which it invariably does if the hammer is wielded properly. Although the instrument is constructed of the finest and hardest steel, it is generally found that more or less has been abraded, and this must either be allowed for or removed, according to circumstances; generally the latter, it being usual to digest the assay in weak hydrochloric acid to remove the steel.