METHOD FOR THE ASSAY OF INDIGO.

By Charles Tennant Lee.

The determination of indigo blue, or indigotin, in indigo, presents various difficulties. The processes in use are long, and subject to considerable error. The methods which depend upon the reduction and subsequent measured oxidation of indigo, require the elimination, previously, of all other reducible bodies, to insure accuracy—an operation both long and tedious. The method by formation of sulpindigotine and its estimation by a standardized permanganate solution, always gives too high results by reason of the presence of other oxidisable bodies.

For several years the author has used a method by sublimation, which has been uniformly satisfactory. Indigo blue sublimes readily and, by a careful regulation of temperature, can be separated from the other components of indigo, indigo brown, indigo red, mucilaginous matter, etc.

The operation is best effected in a shallow platinum tray. Those in use are 7 cm. long, 2 cm. wide, 3-4 mm. deep. Into such a tray is weighed about 0.25 grms. of finely powdered indigo which has been dried at 100° C. The weighing should be rapid to avoid absorption of moisture, and it is best not to exceed this amount greatly for a tray of the size noted, in order that the layer of indigo may be thin.

Spread the weighed powder evenly over the tray by tapping it with the finger; this can be done easily if the bottom of the tray is quite flat, with no rounding towards the sides. Sublime on an iron plate, at first raising the heat gradually to avoid burning.

When the surface of the indigo is covered over with a shining layer of crystals, turn down upon the plate a piece of Russia iron bent into the form of a flat arch, the highest point of which is about 1 cm. above the plate, and a little longer than the tray. Lower the heat at the same time that the arch is put on, as the temperature rises rapidly.

The purple vapors of indigotin are now given off, a portion condensing upon the under sides of the arch. Raise the heat slowly, and enough to maintain a constant sublimation of indigotin. By raising the arch the progress of the work is seen. For a 50% indigo the time required is 30–40 minutes; but soft, Java indigo must be sublimed with more caution, and sometimes require

two hours. The last crystals of indigotin are easily seen upon the the dark colored surface of the residue. When all have disappeared, remove the tray, cool in a dessicator, and weigh. The loss in weight is indigotin. Observe that the heat be no greater than is required to sublime the indigo blue ; and that no yellowish vapors appear, which would indicate the destruction of the residue, leaving only ash.

If the bottom of the tray is flat and everywhere touches the plate, the sublimation goes on regularly, except in case of very rich indigos, already mentioned, when care must be exercised to prevent burning.

Results by this method are constant within $\frac{1}{4}$ of 1 per cent., but the author has frequently made re-determinations with variations of only half that error.

A little practice enables one to leave the sublimation with only occasional attention, and three or four determinations may be carried on at once under the same arched cover.

For commercial and industrial purposes this method appears to have decided advantages. Its rapidity is in great contrast to the other methods which admit of perhaps two determinations in a day, while in point of accuracy it is not wanting.

Laboratory 45 Kilby st.,
Boston, 5th May, 1884.