

took five minutes. It then showed an e. m. f. of 0.0013 volt against pure mercury. Another portion of 300 g. was shaken five minutes in a separatory funnel with some of the same solution. At the end of this time it showed an e. m. f. of 0.23 volt. On shaking five minutes longer the e. m. f. dropped to 0.0104 volt. It is thus evident that the modified L. Meyer method is far more rapid and efficient.

A large quantity, 3 kg. of mercury, was then purified. It had been used for mercury cathode work and was very impure. It was first allowed to stand over night in a large shallow dish containing dilute sulphuric acid. A piece of platinized platinum foil was put into the acid in electrical contact with the mercury. The large overvoltage of hydrogen on mercury prevents the solution of metals from the mercury unless this precaution is taken. This mercury was then run through the apparatus five times, which required twenty-five minutes. It was then tested and showed an e. m. f. of 0.0002 volt against distilled mercury. One more washing, making six times in thirty minutes for 3 kg., yielded mercury which differed from the distilled by less than 0.0001 volt, the limit of sensibility of the electrometer. This corresponds, according to Hulett and Minchin, to less than one part of zinc in ten billion of mercury.

The advantage of the use of cloth instead of the usual drawn out funnel is so obvious that it seems unlikely that the writer is the first to use it.¹ Since, however, it is not recommended by the standard text-books on laboratory methods, it will doubtless be of interest to those handling mercury to know how it may be purified in a very short time and with very simple apparatus, a degree of purity being obtained comparable with that of distilled mercury.

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NOTE.

Quantitative Determination of a Dissolved Substance in Presence of Suspended Material.—It frequently becomes desirable to determine the total amount of some substance in a solution which also contains an unknown amount of a precipitate or other foreign substance. If an aliquot part of the whole volume is taken and the whole amount of substance calculated from the amount found in the part taken, an error is introduced, the amount of which depends on the relation be-

¹ Gooch (*Am. J. Sci.* [3], 44, 239 (1892)) has recommended a scheme whereby a stream of mercury is "atomized" by a blast of air and then allowed to fall into a funnel provided with a gooseneck and containing mercurous nitrate. The apparatus, however, requires some time to construct and adjust, and the globules of mercury fall through only a few centimeters of solution instead of 1.5 m. as in the apparatus here described.

tween the volume of the precipitate and the volume of the whole solution and of the part taken. This error is frequently negligible but in some cases it may be large enough to render the whole determination of little value. The method described below avoids this error at the expense of little additional work. This method consists in filtering or decanting a definite amount of the whole solution, determining the amount of material in the portion taken, making the remainder up to the original volume, filtering or decanting an amount equal to that taken at first, and determining the amount of material in this portion. Then calling the first result m and the second n , the total amount of material in the solution is

$$\frac{m^2}{m-n}$$

This is proved as follows:

Let a = total volume.

p = volume of precipitate.

$a-p$ = true volume of solution.

s = volume taken each time.

x = total amount of material in solution.

$\frac{x}{a-p}$ = total amount of material in 1 cc. solution.

$\frac{sx}{a-p}$ = total amount of material in s cc. solution.

$$\frac{sx}{a-p} = m. \quad (1)$$

$$p = \frac{ma-sx}{m}. \quad (2)$$

$x - \frac{sx}{a-p}$ = amount of material left after taking s cc.

$\frac{x - \frac{sx}{a-p}}{a-p}$ = amount of material in 1 cc. after diluting to original volume.

$\frac{s \left(x - \frac{sx}{a-p} \right)}{a-p}$ = amount of material in s cc. after diluting to original volume.

$$\frac{s \left(x - \frac{sx}{a-p} \right)}{a-p} = n. \quad (3)$$

Substituting (2) in (3) and solving

$$x = \frac{m^2}{m-n}.$$

This result, it may be noted, is entirely independent of a and s , the total volume and the volume taken each time, so there is no advantage in using accurately graduated vessels. Any vessels which can be filled accurately to the same mark each time will serve. Any constant error

in the determination must be carefully avoided or compensated, as the final result will be altered by the square of any such error. The determination may be made by volumetric, gravimetric, optical, or any other method. The above formula has an advantage over the similar one used by Wiley¹ in his well-known "double dilution" method in that only one sample of the original material is required. WARREN RUFUS SMITH.

LEWIS INSTITUTE, CHICAGO,
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[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF NORTH CAROLINA.]

CONDENSATION OF CHLORAL WITH PRIMARY AROMATIC AMINES. III.²

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The other papers upon this subject were published in *THIS JOURNAL* in 1902³ and 1908.⁴ A review of earlier work was given in the second paper and therefore will not be repeated here. Chloral does not combine with all primary aromatic amines. We have been unable to condense it with those containing more than two negative substituents in the nucleus such as trichloraniline. Eibner⁵ noticed this fact, but we gave the reaction a more thorough study. The condensation products with aniline and the toluidines are less stable than those containing one or two negative atoms or groups substituted in the benzene ring. The action of chloral upon the former takes place rapidly with the liberation of considerable heat while with the latter it is necessary to apply heat in order to bring the reaction to completion. Addition products of one molecule of chloral and one molecule of the amine are quite generally possible but we describe here only two which made their appearance in our attempts to prepare the condensation products. These are addition products of chloral with 2-nitro-4-toluidine and 3-chlor-4-toluidine. Such compounds are usually best prepared in ether solution at a low temperature.

The condensation products are resolved into their constituents by heating with strong mineral acids. A special study was made of the action of bromine upon them when dissolved in cold glacial acetic acid. No bromine derivative was obtained in any case, but cleavage al-

¹ *THIS JOURNAL*, 18, 430.

² This paper forms part of a thesis presented to the Faculty of the University of North Carolina in May, 1909, by Stroud Jordan, candidate for the degree of Doctor of Philosophy.

³ Wheeler and Weller, *THIS JOURNAL*, 24, 1063.

⁴ Wheeler, *Ibid.*, 30, 136.

⁵ Eibner, *Ann.*, 302, 370.