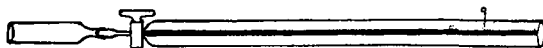


the trouble begins, for the moment we let our absorbing fluids flow in, the narrow neck of the eudiometer is immediately filled with the liquid, and naturally drives the gas down below the zero mark, thus causing much trouble and annoyance. Now Prof. Doremus will say that this difficulty can easily be overcome by first letting pure water run into your burette, then marking the expansion caused, and finally making allowance for this expansion after we begin to absorb our different constituents.

But I will here remark that, as soon as our water level has fallen below zero, it is almost impossible to make accurate readings with the naked eye, which can only be done with a telescope having cross hairs, and such a thing as this for quick work is entirely out of the question.

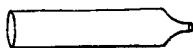
Therefore, to get rid of all this trouble, I have done away with the long neck, and have had a eudiometer made with the glass cock soldered directly on to the top of the burette.



This burette is divided into 100 c.c. and  $\frac{1}{10}$  of c.c., thus permitting me to make very close readings.

The *modus operandi* of this instrument is the same as in the previous case, and the analyses obtained are approximately correct.

The modification here mentioned may be better understood by the following diagram :



### XIII.—1. NOTE ON THE PRECIPITATION AND PURIFICATION OF BARIUM SULPHATE. 2. QUALITATIVE TEST FOR CARBON DISULPHIDE AND CARBON DIOXIDE IN COAL GAS.

By T. O'CONNOR SLOANE, A.M. PH.D.

#### 1.

To obtain a precipitate of barium sulphate that will not run through the filter paper, the following rules should be observed : The solution should be barely acid. This end is best secured by the use of cochineal or some equivalent solution. By using the colored matter in the fluid form, a solution can be neutralized in quarter of the time, and with far more exactness than when its reaction is tried

with litmus paper. The precipitant is added when the solution is nearly boiling, and the liquid is kept at that temperature for some time. A heavy precipitate will be formed with perfectly clear supernatant liquor. It is quite certain, if any iron be present, that some will be carried down, and the ignited barium sulphate will not be white. Many ways of treating the precipitate after ignition have been recommended. I will refer to only two in the hurried way. The first is, to boil it with dilute hydrochloric acid ; the second, to boil it with concentrated sulphuric acid ; then dilute and filter. If the first course be pursued, any barium sulphide present will be dissolved, thus causing a loss. This is quite a common case, as the filter paper reduces the sulphate in the ignition. If treatment with concentrated sulphuric acid be adopted, any other salts of barium present will be rendered insoluble, and excess of weight be caused. In the dry way, fusion with sodium carbonate and a little sodium nitrate, and subsequent determination of the sulphuric acid, may be recommended as very efficacious ; but it is troublesome.

Some time ago I had occasion to make a great many sulphur determinations in coal. I was much annoyed by the precipitation of iron with the barium sulphate. I adopted the following simple method of purification, and it always secured a perfectly white precipitate :

The sulphuric acid was precipitated with great care, as already described, so as to obtain a clear supernatant fluid. The solution was decanted to the last drop through a filter paper. To the precipitate left in the beaker were added five or ten cubic centimeters of concentrated hydrochloric acid. The beaker was then carefully held over and just touching a hot plate, until the acid was in full ebullition. By holding the beaker in an inclined position there will be no danger of spirting. The boiling was kept up for about a minute. After cooling, it was diluted, cochineal solution added, overneutralized with ammonia, slightly acidified and filtered. This always gave perfectly white precipitates.

2.

A piece of caustic potash a few mms long is dissolved in 10 to 20 c.c. of alcohol. If, after standing protected from the air, a homogeneous and clear solution remains, it is ready for use. Otherwise, after settling, the clear fluid may be decanted. It is placed in a suitable absorption tube and a cubic foot or more of gas passed through it. It is then poured into a test tube. If the gas contained carbonic acid, an oily-looking colorless solution of potassic carbonate

in water will underlie the alcohol. This last will be colored more or less.

The alcoholic solution, if any carbon disulphide was in the gas, will contain a so-called xanthate of potash, and may be boiled and tested for sulphuretted hydrogen.

A more demonstrative method is to add excess of copper salt, filter out the precipitated cupric compounds, and pour ammonia through the filter paper, when a highly characteristic precipitate of cupric xanthate will remain on the filter paper.

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#### XIV.—UPON THE DIRECT CONVERSION OF THE AROMATIC AMIDES INTO THEIR CORRESPONDING AZO-COMPOUNDS.

BY ALBERT R. LEEDS.

I have already published in another place (JOURNAL AMERICAN CHEMICAL SOCIETY, 3, 16,) the results obtained by the action of hydrogen peroxide upon benzene. The latter is gradually converted into phenole-oxalic acid, and a small amount of a yellow coloring matter being formed at the same time.

Phenole itself is but slightly acted upon by hydrogen peroxide in the cold, but at the boiling point undergoes conversion into a tarry substance.

Naphthalene, when digested with hydrogen peroxide, yields a small amount of naphthole. Naphthole itself is not affected to any notable extent by hydrogen peroxide in the cold, but at the boiling point is changed to a tar.

When anthracene is dissolved in glacial acetic acid, and the precipitate obtained on the addition of solution of hydrogen peroxide is digested with excess of the latter re-agent, the anthracene is slowly converted into anthraquinone. Three grms of anthracene, boiled for three days with hydrogen peroxide, in a flask attached to a return-cooler, were changed almost entirely into anthraquinone, the brown coloring matter formed at the same time being too small in amount to permit of its being experimented upon successfully.

Benzenesulphonic acid was slightly acted upon, a brown color being imparted to its solution after boiling for a number of hours with hydrogen peroxide, and the acid crystallising out from the liquid in small colorless crystals, identical in appearance with the original body before its treatment with peroxide.

The preceding results show that in certain cases hydrogen peroxide operates upon organic bodies by the substitution of hydroxyl ; in other cases, by direct oxidation, and in the third place, by both actions combined.