114. *A Constant Sulphite Solution.*

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This paper sets out a method for the preparation, storage, and manipulation of a sulphite solution which will remain of constant value for an indefinite period of time.

MACAULAY (J., 1922, **121**, 552) kept a sulphite solution constant by maintaining an atmosphere of carbon dioxide in the store-bottle, but for long storage traces of certain gases should be removed from the carbon dioxide. This may be done by generating the gas from marble and dilute hydrochloric acid and passing it through a series of scrubbers containing (1) arsenious oxide in sodium hydrogen carbonate solution, (2) cuprous chloride in hydrochloric acid solution upon copper gauze, (3) sodium hyposulphite in potassium hydrogen carbonate solution upon iron gauze, and (4) a mixture of crystals of ferrous sulphate and of sodium carbonate decahydrate which, of course, soon becomes lumps of sodium hydrogen carbonate.

A store-bottle containing dilute hydrochloric acid which has been boiled free from air and saturated with carbon dioxide feeds this main generator and also a smaller similar one (also fitted with scrubbers), which acts as an auxiliary and maintains an atmosphere of purified carbon dioxide, not only in the main generator, but also in itself and in the store-bottles for dilute hydrochloric acid, cuprous chloride and sodium hyposulphite solutions, and the jacket surrounding the nozzle of the pipette used to measure out the sulphite solution. The store-bottles for the cuprous chloride and the sodium hyposulphite solutions are kept connected to their respective scrubbers by glass tubes with taps so that they may be flooded periodically.

The sulphite store-bottle, a 6-l. Pyrex flask, is filled with distilled water, which is then boiled and cooled in an atmosphere of carbon dioxide. The flask is now fitted to the main carbon dioxide generator, and its contents stirred for some days in a slow stream of the gas. The desired quantity of sodium sulphite is then added, and after a day or so stirring is resumed until titrations show constancy.

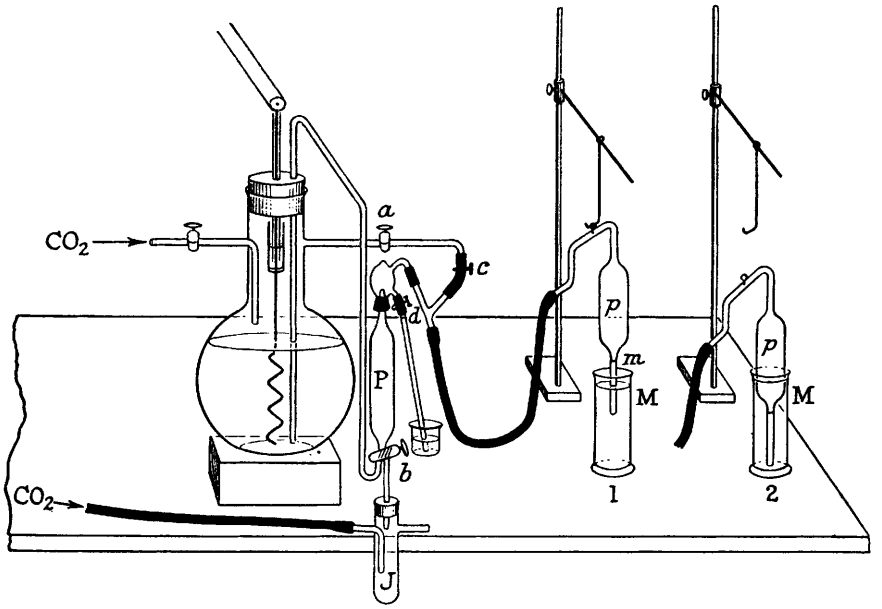
Experiments with constant sulphite solution are usually arranged so that the contents of an automatic pipette, which is part of the apparatus and works with an atmosphere of purified

carbon dioxide, are used for each experiment. Vessels filled with carbon dioxide gas are used to receive the contents of the pipette.

The following figures illustrate the constancy of the sulphite solution :

Date, 1937.	Temp.	Iodine soln., g.	Sulphite soln., g.	$Na_2S_2O_3$ soln., ml.	$Na_2S_2O_3$ equivalent to sulphite, ml.	Available SO_2, g.
Aug. 6	18·22°	51·065	50·232	11·31	37·25	0·12016
„ 6	18·25	51·060	50·238	11·39	37·17	0·11990
Sept. 3	16·00	51·080	50·256	11·40	37·16	0·11987
„ 3	16·00	51·100	50·256	11·45	37·11	0·119705
„ 10	16·00	51·0875	50·280	11·40	37·16	0·11987
Aug. 5	18·30	51·085	—	48·51		
„ 5	18·30	51·080	—	48·55	mean = 48·56	
Sept. 11	15·50	51·075	—	48·60		
„ 11	15·50	51·090	—	48·55		

To ensure a regular delivery from the automatic pipette under approximately atmospheric pressure finally, the arrangement shown in the figure is used.



Filling the automatic pipette P. This pipette is filled by closing the stopcock *a* and opening the three-way stopcock *b* so that the liquid syphons into the pipette *P* and floods it above the zero mark; *b* is then closed.

Emptying the pipette P *under approximately atmospheric pressure.* The stopcock *a* is opened so that the gas, still controlled by screwcock *c*, bubbles slowly from the pipette *p* (100 ml.). The water in the cylinder is then adjusted to a fixed mark *M*, and *a* is closed. When the pinch-cock *d* is opened, the overflow in *P* runs off and the water in the cylinder rises in *p* until the zero of *p* is passed, whereupon *d* is closed. The liquid in *p* is then brought down to its zero mark *m* by opening *a* cautiously and finally closing it. Any overshooting of the zero *m* may be readjusted by means of the pinchcock *d*; *p* is now disconnected from its hook and lowered into the cylinder (position 2). The carbon dioxide jacket *J* is then removed from the nozzle of *P*, stoppered, laid aside, and replaced by a vessel containing carbon dioxide. The contents of *P* are run into the vessel, and then *J* is replaced. The vessel, now containing constant sulphite solution, is weighed if necessary, and the sulphite used as required. The pipette *p* is then lifted on to its hook (position 1) and the head of liquid in it is available as an aid to the easy refilling of *P*.

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