carbon and calcium oxide. The uncombined carbon was determined by treating the sample with dil. nitric acid, filtering off the carbon and weighing. The difference between the uncombined carbon and total carbon, obtained by combustion, represents carbon combined as carbide. These values are recorded in the last column of Table II.

TABLE II

ANALYSES OF SAMPLES

CaC_2 %	C as CaC_2 %	Total C minus uncombined C %
72.08	27.01	27.02
66.47	24.91	24.97

It will be seen that the agreement obtained for carbide carbon by the two methods is satisfactory.

Summary

An accurate method has been developed for the determination of carbides that yield acetylene when treated with water. Traces of sulfides do not interfere when proper precautions are taken. Phosphides, however, should be absent.

WASHINGTON, D. C.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF CALIFORNIA]

A SIMPLE PRESSURE-MEASURING DEVICE FOR USE WITH CORROSIVE GASES

BY DAVID F. SMITH[1] AND NELSON W. TAYLOR

RECEIVED MARCH 17, 1924

Perhaps the simplest and most satisfactory glass pressure-measuring device heretofore described is that of Daniels and Bright[2] or the later modification described by Karrer, Johnston and Wulf.[3] Our experience in the use of these devices has brought out several inherent disadvantages in them, however. In the first type it is difficult to make the diaphragm thin enough to be sensitive and at the same time thick enough to avoid breakage in use or distortion in the process of burning in the platinum. The platinizing process is itself very troublesome. Many otherwise good pieces are spoiled by faulty platinizing. The adjusting of the contact points is troublesome and often results in damage to the diaphragm; and eventually the diaphragm becomes useless on account of the wearing off, burning by the current, or smutting of the platinum at the contacts. Moreover, the whole process of setting up the pieces must be gone through in order to determine whether the diaphragm is suitable.

[1] National Research Fellow in Chemistry.

[2] Daniels and Bright, THIS JOURNAL, **42**, 1131 (1920).

[3] Karrer, Johnston and Wulf, *J. Ind. Eng. Chem.*, **14**, 1015 (1922).

The second type is better, inasmuch as it eliminates the platinizing and its attendant difficulties. However, the labor in making and trying out the diaphragms is nearly as great. When Pyrex glass is used the platinum seals are not satisfactory. Temperature changes throw the apparatus out of adjustment and the adjustment itself is very troublesome, and indeed impossible when the diaphragm is enclosed, as is often the case, in a more or less complicated piece of apparatus. The platinum contact points cause trouble unless they are kept scrupulously clean. Either type of device is thus tedious to make, easily gets out of order, and has limited sensitivity. The use of sulfuric acid manometers, protective coatings over mercury, etc., is fairly satisfactory in some cases, but in working at temperatures higher than room temperatur there is frequently trouble because of distillation from the hot sample tube into the cold manometer.

The apparatus described herein was made many months ago by the first-named author of this paper, and subsequently duplicates were made by both of us and were tested by us jointly.

The new device is made by blowing a fairly thin bulb on the end of a 4–8mm. glass tube and flattening the end of the bulb, but during the flattening, so directing the flame that a small "wrinkle" or uneven surface results. Change of pressure then causes a "clicking" noise as the uneven glass diaphragm passes a critical position, once as the pressure is directed outward and once when it is directed inward. Both of us have made several of these devices (of Pyrex glass) ranging from 10 to 23 mm. across the flat part. It is unnecessary to make the diaphragm excessively thin. We have used several which were strong enough to withstand an atmosphere of pressure in either direction and were still sensitive to 0.2 mm. The pressure difference between the two "clicking" points ranged from 1 mm. to 100 mm., the reproducibility of the separate critical points being not greatly dependent upon this range in pressure. For example, several pieces we have used were capable of withstanding a pressure difference of one atmosphere, had a zero correction of 25–35 cm. and a pressure range between critical points of 10 cm.; the separate critical points were, nevertheless reproducible to 0.2 mm. or less. The pieces having large zero corrections usually make the loudest noise upon passing the critical points.

The method of mounting is shown in Fig. 1.

A is the diaphragm itself, enclosed by a slightly larger tube. B is the sample tube containing the corrosive gas whose pressure is to be measured. This whole part of the apparatus can be enclosed by a constant temperature bath. F is an ordinary mercurial manometer communicating through the air in the interior of the apparatus with the upper side of the glass diaphragm. E is a 3-way stopcock establishing communication either with reduced or elevated air pressure or with the small tube and leveling bulb D which permits small pressure changes to be made inside the apparatus. When diaphragm A is exposed in the room the click is audible 6 meters away. When it is en-

closed as shown, the sound is deadened somewhat. Although coming from inside the apparatus, the click is nevertheless in most cases clearly audible, even down to pressures as low as 10 cm. If it is desired, a telephone transmitter C may be placed inside the apparatus on the "air side" and connected through two-stage vacuum-tube amplifiers with a pair of receivers. The tube to hold the transmitter is made by sealing to the bottom of a Pyrex beaker, as shown, a tube connecting with the manometer line and two capillary tubes in which to seal by means of wax the transmitter wires. The transmitter is best laid upon cotton to deaden extraneous noises and the beaker is finally cut off and sealed with wax to a glass or metal plate. If it is desired to avoid using the electrical equipment, a pair of glass or metal diaphragms held over the ears and connected to the manometer line by means of a piece of large, rubber pressure-tubing (in the fashion

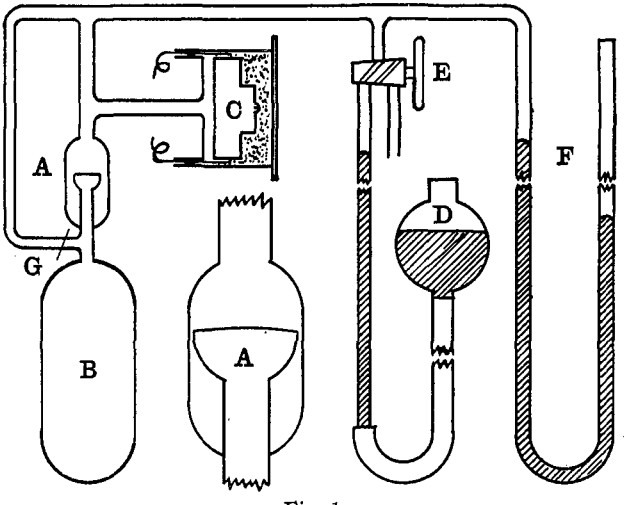

Fig. 1.

of a diaphragm stethoscope) has been found satisfactory at least down to pressures as low as 15 cm. We have found it quite easy, however, to make pieces with a response loud enough to be heard outside the apparatus by the unaided ear, even when there was considerable noise both in the apparatus (from a boiling liquid) and in the room (from vacuum pumps). The side tube G may be used at the start to keep the pressure equalized on both sides of the diaphragm and is later sealed off at the point indicated.

We have made many tests upon various pieces and have always found that the zero correction and reproducibility of the clicking points were in-

TABLE I

REPRODUCIBILITY

Test 1						
Actual pressure, mm.....	740.0	1064.0	364.0	0.0	...	...
Measured pressure, mm..	(764.0)	1064.2	364.2	0.1	...	...
Test 2						
Actual pressure, mm....	764.0	242.2	242.2	242.2	114.4	114.4
Measured pressure, mm..	(764.0)	242.3	242.1	242.2	114.8[a]	114.4

[a] Poor meniscus.

dependent of the pressure. The results of two typical tests made by connecting manometers to both sides of the diaphragm, are given in the tables labelled Test 1 and Test 2.

Neither of the diaphragms used was excessively thin. The one used in Test 2 was capable of withstanding an outward pressure of at least 60 cm. No special care was taken in this test to have the manometer tubes or mercury scrupulously clean nor to check the cathetometer used. However, it is readily seen to be very easy to make diaphragms of this type which are sensitive to at least 0.2–0.3 mm. Furthermore, we have used diaphragms which had been "clicked" probably hundreds of times with no damaging results to them. We have used these devices at temperatures from 20–360° and have observed no change in their behavior within this temperature range.

Although the blowing of the diaphragms is a more or less "cut and try" process, several satisfactory pieces can be turned out within an hour or so after a little practice. It is possible to sort out the satisfactory pieces very easily—merely by exerting pressure in the blowing tube. The setting up of the satisfactory pieces is all simple glass blowing.

Summary

1. An accurate, and easily made all-glass device for measuring the pressure of corrosive gases has been described.

2. Tests showing the accuracy attainable with the device have been presented.

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[CONTRIBUTION FROM THE COLORADO SCHOOL OF MINES]

DIPHENYLAMINE INDICATOR IN THE VOLUMETRIC DETERMINATION OF IRON

BY WILFRED W. SCOTT

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The excellent method developed by J. Knop[1] using diphenylamine as an internal indicator in dichromate titrations of iron, makes one wonder why it is that this was not thought of before. The writer suggests an application of the same principle to iron titrations with potassium permanganate. The advantage in the use of diphenylamine over the use of permanganate alone is in the fact that titrations may be made in the presence of mercurous chloride, the end-point being sharp and permanent, furthermore, the blue color or violet, as the case may be, is more pronounced than the pink of permanganate alone. Hydrochloric acid does not interfere in concentrations ordinarily present. The use of stannous chloride for reducing iron has advantages over zinc, and this procedure for reduction

[1] Knop, THIS JOURNAL, **46**, 263 (1924).