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SUBJECT The Analysis of C-216

Report from Hooker Electrochemical Co. (100 XR-4245)

TO File

FROM L. E. Tufts and R. H. Kimball

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THE ANALYSIS OF C-216

Research Analytical Laboratory
Hooker Electrochemical Company

The fluorine gas passes through a nickel tube filled with dry NaCl mixed with 7% of NaF, which converts the fluorine to chlorine and absorbs the HF. The converted gas is then analyzed by the method described in the section "The Analysis of Gas from the Hooker Type S Chlorine Cell".

A - Apparatus

The conversion tubes are diagrammed in Figure 3. A and B are U-tubes made from 3/4" seamless nickel tubing, capped with 1/2" nickel unions, machined to fit the tubing. The manifold C is made of standard 1/4" brass fittings, and is attached to the C-216 line through a length of 1/4" copper tubing D with tube couplings. A glass adapter E is attached to the discharge side with a rubber connection, and serves as a connection for the gas sampling apparatus shown in Figure 2.

Valves 1, 2, and 3 are standard 1/4" Hoke brass needle valves. Before using for the first time, the empty equipment is assembled and well conditioned by exposure to C-216 gas.

B - Preparation for an Analysis

The filling used in the U-tubes is C.P. NaCl mixed with 7% of C.P. NaF. Individual charges of 120 grams NaCl mixed with 8 grams NaF are dried in the oven at 150°-200°C. Tubes A and B are heated in the oven and then filled with the dry mixture, using 128 grams in each tube. Each is then theoretically capable of converting over 20 liters of C-216, which is about three times the actual requirement. After filling, the tubes are replaced in the oven for one-half to one hour, then quickly connected up while still hot, and a stream of dry air is blown through the entire apparatus while it cools. The air is dried by passing over a layer of KOH followed by anhydrous magnesium perchlorate (Anhydrone), in a 1" Pyrex tube 30" long. If desired, tightness can be tested at this stage by evacuating the system against a manometer, protecting it from moisture and releasing the vacuum with dried air. Both ends of the apparatus are protected by rubber nipples until it is attached to the C-216 system.

C - Procedure for Sampling

Be sure that valves 1, 2, and 3 are closed. Connect the copper delivery tube D to the sampling line, by means of a suitable tube coupling and adapter. To connect E with the sampling apparatus, use the rubber covered adapter designed for the use of slight vacuum, and described on Page 3 of the following section on the analysis of chlorine cell gas. Connect the extra length of glass tubing on the delivery end of the adapter to an empty 500 ml suction flask, which serves as a trap. This in turn is connected to a 6 mm tube reaching to the bottom of a second 500 ml suction flask, which contains water and serves to show the rate of flow of gas through the conversion tubes while purging. This suction flask is connected to a convenient source of mild suction, such as a water aspirator, and carries a vacuum break to permit easy regulation of the degree of suction applied. These directions are restricted to the sampling of C-216 from the cell, where the use of mild suction is necessary.

The gas sampling apparatus (Figure 2) described in the second section should be filled and ready for use.

When the conversion tubes are connected as described, purge them as follows. Open the vacuum break, and turn on the suction. Open the screw clamp in the capillary adapter, and open valves 1 and 2 so as to draw C-216 through the whole system. Now gradually close the vacuum break, which slowly increases the suction, until gas is flowing through the conversion tubes at the rate of 2 to 3 bubbles per second in the bubbling flask.

The C-216 is converted to chlorine in tube A, which should immediately become hot at the inlet end. The converted C-216 purges the remainder of the apparatus. The flow should be kept slow enough to prevent the reaction area of the conversion tube from becoming too hot, which would result in violent attack by the C-216. A practical control is to touch the hot area frequently with a wet rag; the water should steam off, but should not sizzle.

When the "hot spot" in tube A has progressed to a point where about one-third of the filling in tube A has been used up, the apparatus has been sufficiently purged.

Slowly reduce the vacuum on the bubble flask, until no gas is passing through the apparatus. Close the screw clamp at E, and disconnect the 8" length of glass tubing, first from the trap, and then from the rubber covered capillary tube. Quickly connect the gas sampling apparatus (Figure 2) in its place. Close valves 1 and 2, and open valve 3. Open the screw clamp at E. Lower the leveling bulb on the gas sampling apparatus as far as possible, and open the screw clamp on the inlet tube. It may be necessary to apply a little extra suction to the soda-lime guard tube at the top of the leveling bulb, to keep a steady stream of gas flowing into the gas sampling tube; if this flow is interrupted, solution may back up into the capillary and lead to plugging.

The C-216 is now being converted in the fresh U-tube B, which also absorbs HF. As before, the flow should be kept slow enough to prevent the reaction area from becoming too hot. The chlorine and carbon dioxide in the converted gas are absorbed by the alkaline arsenite, and the residual gases collect in the sample tube. Depending upon the purity of the C-216, continue the absorption until 25-50 ml of residual gas has collected.

Close the screw clamps on the inlet tube and at E, and the valve 3. Detach the gas sampling apparatus, which is now ready for the analysis of the residual gases. Connect the capillary tube again to the trap and bubble flask as before. Open valves 1 and 2, and draw C-216 through again for 5 minutes. This serves to sweep the C-216 left beyond valve 3 into tube B.

Close the screw clamp at E, and close valves 1 and 2. Disconnect the capillary from the trap, and disconnect the copper tube D from the sampling line. Do not replace the rubber cap on tube D while it is filled with C-216.

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D - Analysis of the Residual Gases

Analyze the residual gases, titrate the alkaline arsenite solution for chloride, and determine carbon dioxide by the evolution method, as described in the second section dealing with the analysis of chlorine cell gas. The volume of fluorine in the sample at STP is considered to be the same as that calculated for the chlorine from the chloride titration. The remaining calculations are carried out as described for chlorine.

E - The Determination of HF

The HF in the sample absorbed for analysis is retained as the acid fluoride in tube B. It can be determined by dissolving the tube contents in excess 0.5N $NaOH$, and titrating the HF. It is very difficult to free the tube contents of free chlorine, even by long blowing with dry air. We have therefore assumed that any free chlorine remaining will be converted to chlorate during solution of the tube contents in hot caustic and have corrected for it by determining chlorate in the solution. Experiments are now under way to find a simpler and more satisfactory method of avoiding this possible source of error.

L E Turts
L E Turts

January 25 1944

R H Kimball
R H Kimball

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CONVERSION TUBES

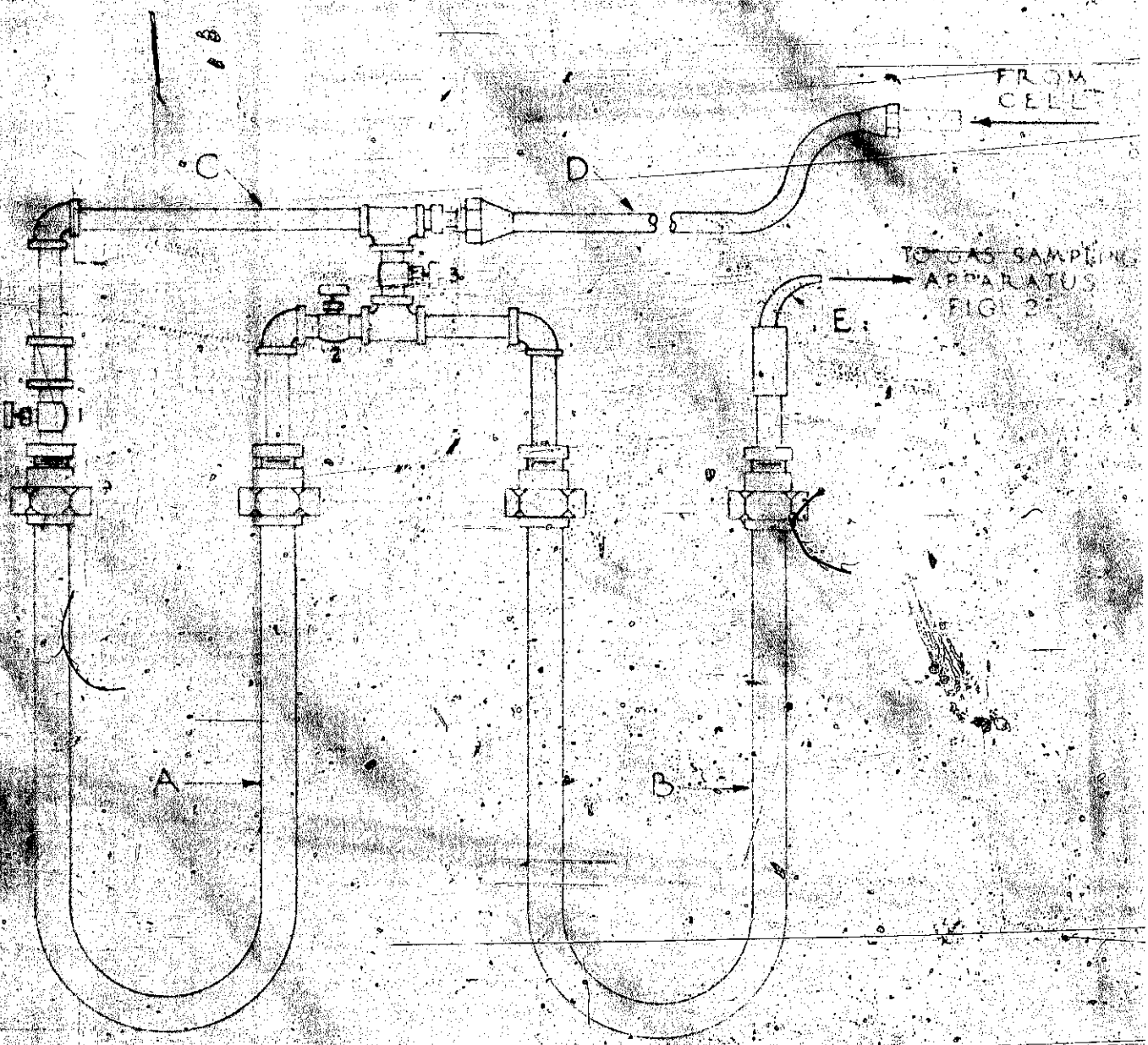

Figure 3