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MODIFICATION OF A CARBON-HYDROGEN-NITROGEN ANALYZER FOR REDUCTION OF INSTRUMENT BACKGROUND

by

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Carbon, hydrogen, and nitrogen (CHN) auto analyzers are becoming more accepted as analytical tools for the determination of hydrogen in inorganic materials (1-3). This acceptance is due in part to the modern, state-of-the-art CHN instruments equipped with autosamplers, multielement capability, sensitive detectors, and automatic data processors. Most CHN analyzers derive the hydrogen percentage of the sample by a direct proportion between element concentrations and the thermal conductivity detector response signals, after blank subtraction. At low concentrations of hydrogen the easiest way to increase the signal to background ratio is to increase the amount of sample being analyzed. However, in the event of limited sample size, an alternative means of increasing the signal to background ratio is to attenuate the background. By a simple modification of a Model 240X CHN analyzer (Control Equipment Corporation, Lowell, Mass.), we have achieved significant improvements in the background.

In a model 240X analyzer, the background signal is caused by both a desorption of water from the dead volume in the autosampler drive box assembly (4), and from a hydrogen memory effect in the combustion train due to residual gases in the analyzer from previous samples. The latter has been studied by D. Elliott (5) and he has recommended matching hydrogen concentrations of standards and samples as a way of coping with the memory effect. To minimize the deleterious effect of dead volume in the autosampler drive box assembly, we redirected the helium carrier gas to pass through the drive box assembly. By continuously purging the drive box with helium, back diffusion of gases into the dead volume is eliminated and the background signal is reduced.

Modification Procedure

As illustrated in figure 1, disassemble the uppermost purge valve inlet on the drive box [A] and connect a 30 in. long 1/16 in. o.d. stainless steel tube [B]. From fittings [C] and [D], disconnect and remove existing 3 in. u-shaped stainless steel tube [E]. Feed the 30 in. tube through existing holes [F] and [G], and connect to the helium supply fitting [C]. Use a 1/4 in. plug to seal off the remaining port hole [D].

Discussion

Prior to the modification, the desorption of water from our 240X autosampler drive box assembly is shown in figure 2. This was demonstrated by running a full suite of 64 blanks and repeating the automated procedure the next day. Also shown in figure 2 are the results of running 40 blanks after the modification. After the modification, we obtain a more linear baseline after only five blank runs and a lowered absolute value for the instrument blank. Typically we condition the instrument by running five blanks prior to analyses of samples. Subsequent to this conditioning step, the blank value using the modified instrument is approximately seven to ten times lower.

Conclusion

By purging the drive box assembly with helium, the hydrogen signal to background ratio can be increased in order to optimize the results for low levels of hydrogen.

Literature cited

1. Skinner, N., Brown, F., and Flanagan, F., Geostandard Newsletter, 5, no.1 (1981) 3.
2. Din, V., Jones, C., Chemical Geology, 23 (1978) 347.
3. Kirsten, W., Analytical Chemistry, 51, no. 8, (1979) 1173.
4. Reed, A., Oral communication, Control Equipment Corp. 1981.
5. Elliott, D., Chemical Automation Update, 1, no. 1 (1984).

FIGURE 1. INSTRUMENT MODIFICATION - TOP AND BOTTOM VIEW

FIGURE 2. INSTRUMENT BACKGROUND - BEFORE AND AFTER MODIFICATION

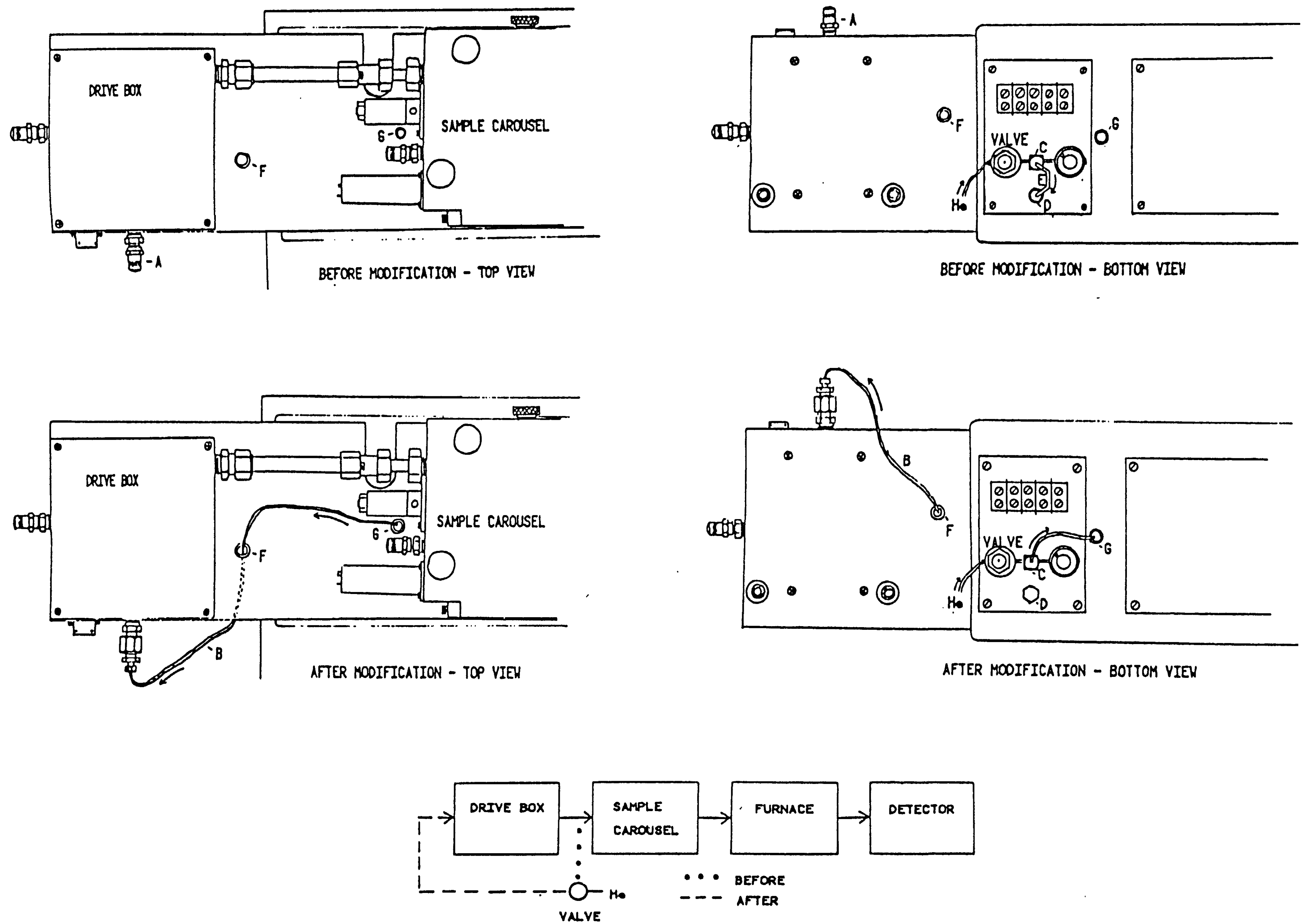
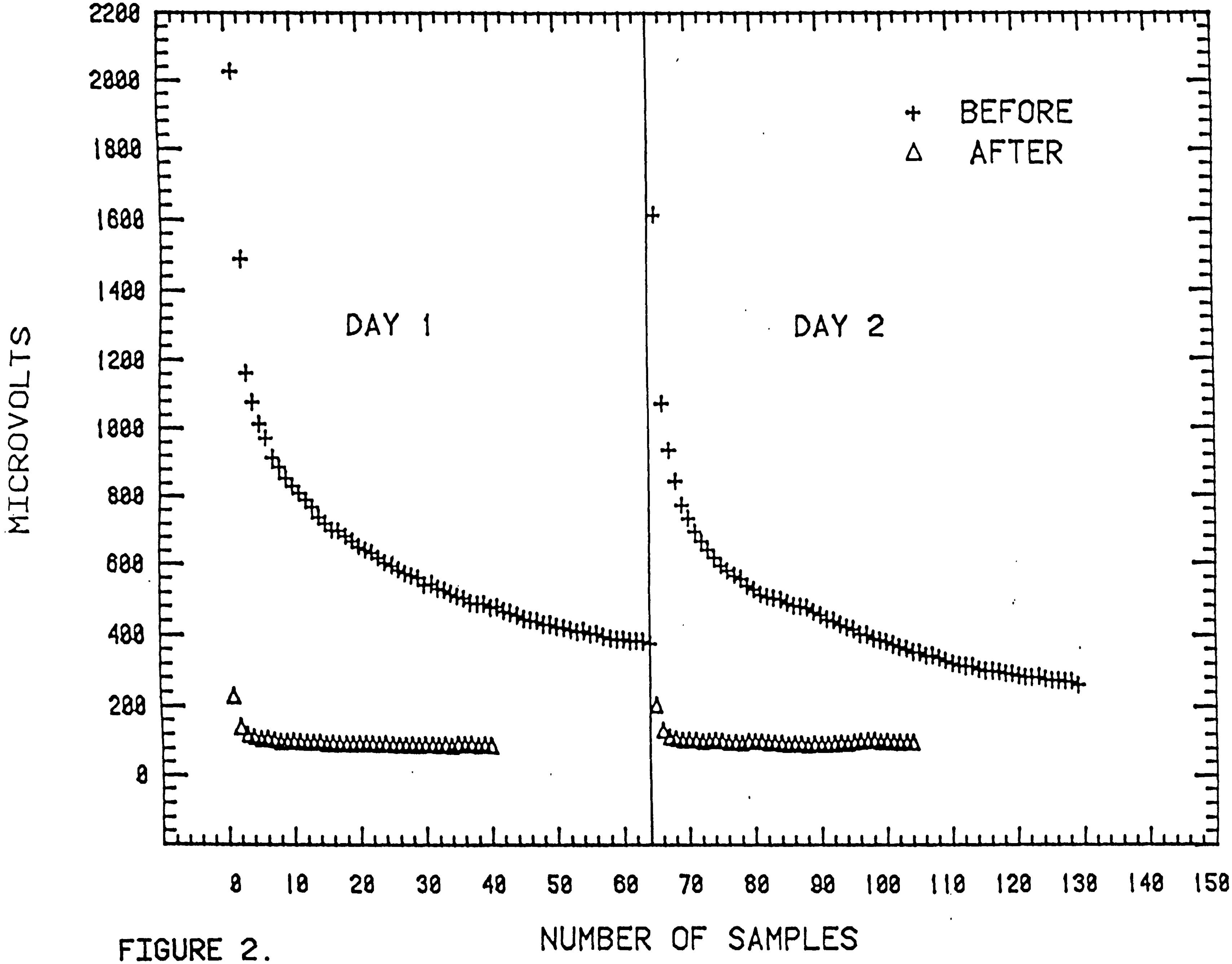

DRIVE BOX
SAMPLE CAROUSEL
A
F
G
BEFORE MODIFICATION - TOP VIEW
VALVE
C
D
E
He
BEFORE MODIFICATION - BOTTOM VIEW
B
AFTER MODIFICATION - TOP VIEW
AFTER MODIFICATION - BOTTOM VIEW
DRIVE BOX
SAMPLE CAROUSEL
FURNACE
DETECTOR
He
VALVE
BEFORE
AFTER

FIGURE 1.



MICROVOLTS
NUMBER OF SAMPLES
DAY 1
DAY 2
+ BEFORE
Δ AFTER
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150

FIGURE 2.