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Note

Sensitivity enhancement of the Coulson conductivity detector by modification of the bridge circuitry

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The Coulson electrolytic-conductivity detector has proven to be a valuable aid for the selective analysis of nitrogen- or halogen-containing compounds by gas chromatography (GC). Much work has been done on the optimization of the system by introducing such changes as PTFE transfer lines[1], smaller-diameter quartz tubes[2], higher furnace temperatures[3], optimization of gas flow-rates[3,4] and water flow-rates[5] through the detector cell, as well as cooling of the detector system[6]. However, the main disadvantage of the detector (even when fully optimized as mentioned above) is still a lack in sensitivity when compared to other GC detectors. The purpose of this note is to report a modification of the Coulson detector by which sensitivity is increased three- to fourfold.

Since the detector cell was already attached to a 1.0-mV recorder, it was decided to attempt to increase detector sensitivity by modifying the conductivity bridge circuitry to permit a larger d.c. voltage across the electrodes. An increase in detector response was observed[2] when the voltage was increased to 54 V from 30 V. Results of varying the voltage up to 100 V d.c. are reported herein. The change in electrical circuitry is simple, and inexpensive. It should prove to be a helpful modification to this useful detector.

EXPERIMENTAL

The conversion of the bridge circuitry to permit up to 100 V across the electrodes is a simple operation. A schematic of the bridge circuit indicating the modifications is shown in Fig. 1. These alterations convert the voltage attenuator from 0–100 V d.c. in 10-V increments (from 0–30 V in 3-V increments). Exact output voltage was verified at each increment by a vacuum tube voltmeter.

GC conditions were as follows. A Microtek MT 220 gas chromatograph (Tracor) equipped with a Coulson detector in the nitrogen mode was employed. The column consisted of 3% OV-1 on Chromosorb W-HP (80–100 mesh) packed in a 1.3 M × 6 mm O.D. glass U-tube. Operating parameters were: column temperature, 195°; helium carrier flow-rate, 60 ml/min; hydrogen flow-rate, 50 ml/min; pyrolysis furnace temperature, 800°. The water flow-rate through the detector was optimized by insertion of a 0.003-in. stainless-steel wire into the capillary water entrance to the

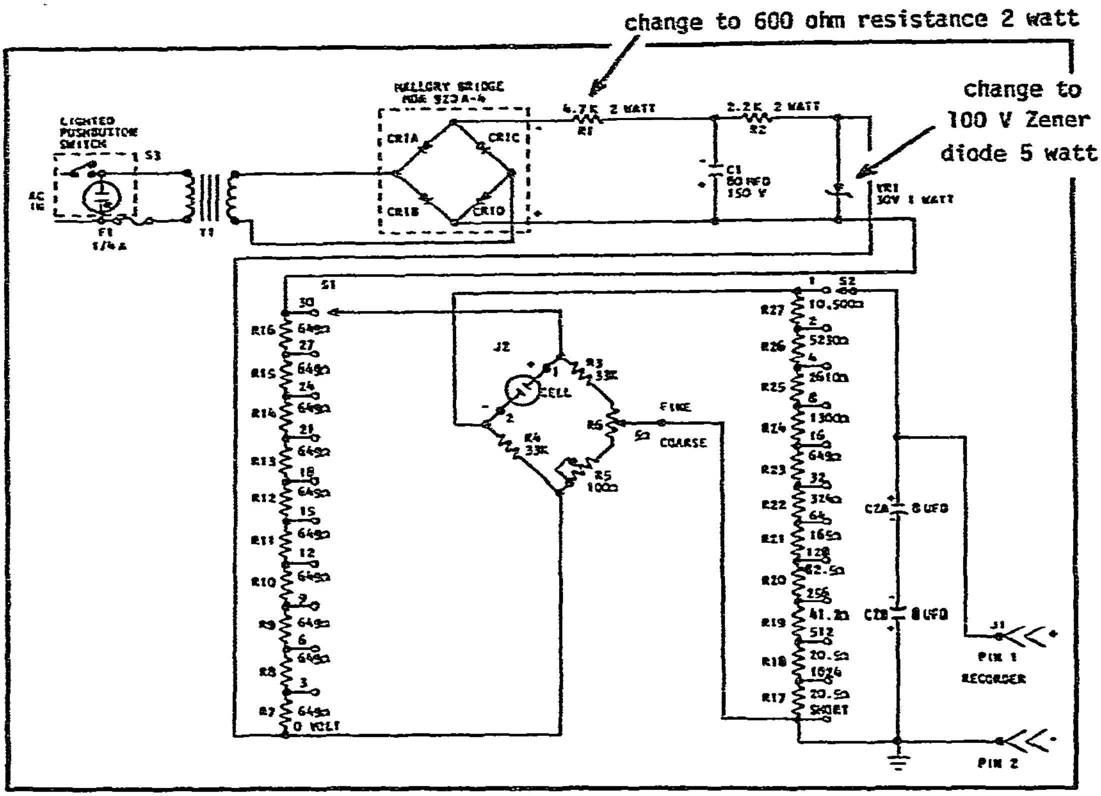

Fig. 1. Schematic diagram of the Coulson conductivity bridge showing modifications to circuitry.

mixing chamber[5]. The herbicide atrazine (2-chloro-4-ethylamino-6-isopropylamino-*s*-triazine) was used as the test compound. The background conductivity was permitted to stabilize for each cell before the voltage study was carried out. A 1.0-mV strip-chart recorder was used for recording the chromatograms.

RESULTS AND DISCUSSION

A plot of peak height *versus* applied voltage for 10 ng of atrazine (8 × attenuation) showed that the increase in sensitivity was linear with voltage. In the chloride mode a similar change with voltage was observed. These results differ from those reported earlier[2] where a pseudoparabolic response was found with increasing voltage (0–54 V). A similar curve was obtained initially in this work, but examination of the voltage at each increment on the voltage attenuater showed that they were not uniform. However, when actual voltages were plotted, a straight line was obtained. Operation at 100 V for extended periods produced no electrolysis, etc. in the system. The maximum output current from the electrodes at 100 V was found to be 6.6 mA compared to 2.1 mA at 30 V.

Fig. 2 compares the results obtained for 5 ng (8 × attenuation) at 30 V and 100 V. The detector is used regularly in our laboratory at 1 × attenuation with 100 V. At this sensitivity 0.8 ng of atrazine produced a 50% full scale deflection. Voltages greater than 100 V were not studied, although use of these should be possible if Coulson stability is very good. Two other Coulson cells were examined, both of which were noisy under normal Coulson operating conditions. Thus, no real advantage

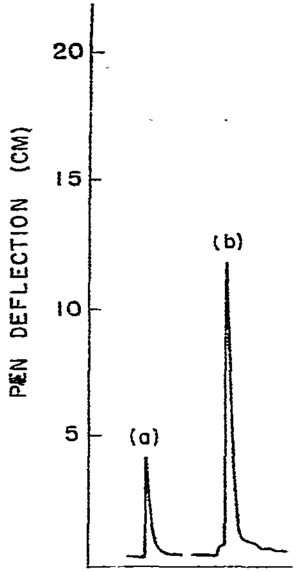

Fig. 2. Comparison of responses at (a) 30 V and (b) 100 V. 2-min retention time for atrazine (5 ng, 8 × attenuation). 1 mV full scale.

was obtained by increasing voltage, since background noise was increased as well. The important point to note, however, is that additional sensitivity can be used when the noise level is low, a feature which is not present on the existing apparatus.

The GLC–Coulson system used had over 100 sample extracts (food, foliage, soil, water) passed through it before this study was commenced. Only the strontium hydroxide scrubber (nitrogen mode) was replaced. Thus the above results were obtained under practical analytical conditions. A newly conditioned GC–Coulson system could provide better peak shape, more stable baseline, and better sensitivity than obtained herein.

CONCLUSION

The use of this modified bridge would be a definite advantage for analysts using the Coulson, since the lack of sensitivity is its major drawback. The change costs only a few dollars in parts and can easily be accomplished. Also this would still permit analyses to be carried out at 30 V, if desired, simply by setting the voltage dial in the appropriate position. The alteration simply allows a greater degree of freedom in the selection of optimum voltage.

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