TOTAL CARBON SYSTEM AUTOMATION


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For:
Harbor Branch Foundation, Inc.

Summer Intern Program
TECHNICAL REPORI' \#29
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## ABSTRACT

A working model was devised and constructed which automates the operation of the total carbon system (TCS) manufactured by Oceanography International. The TCS is a system which measured the total carbon content of sealed seawater samples. Manual operation of the TCS required a technician be at the machine for up to five hours. The automation system frees the technician from this requirement.

To devise and construct a system to automate the TCS analyzer. This system should perform the tasks now requiring the constant presence of a technician with a minimum of supervision and without sacrificing any accuracy of data or destroying any samples.

The system should be separate from the existing manual operation, thus enabling the $T C S$ to be run either manually or automatically. The interphase should be made with a minimum of modifications to the TCS.

The system should be powered by the existing available gas pressure and/or llov AC.

### 1.0 BACKGROUND

### 1.1 INTEREST IN CARBON CONTENT

As a part of Harbor Branch Foundation's on going ten year study of the Indian River ecosystem, it is of particular interest to the chemjstry department to determine the total carbon content of this water. Not only is the organic carbon content a function of the river's productivity, but also extensive research is under way in determining levels and flux of trace elements, since many are organically bound.

### 1.2 DETERMINING CARBON CONTENT

Samples from the Indian River are digested in the presence of an oxidizer. This converts any carbon into $\mathrm{CO}_{2}$. The samples are then sealed in special ampules. At some later time, these ampules (typically 50-80) are individually analyzed by "The Total Carbon System" (TCS), a device manufactured by Oceanography International Corp. The process involves crushing the top of each ampule and running the dissolved $\mathrm{CO}_{2}$ through a LIRA infra-red analyzer (see PROBLEM) where it can be quantitatively analyzed. This process requires that a technician be at the machine for up to five hours.

### 1.3 PRELIMINARY DESIGN OF TCS AUTOMATION

In an effort to find a way of freeing the technician from these hours of operation, Gary Peterson (Chemistry, HBF) sought the help of John K. Holt (Project Engineer, HBF).

After explaining the problem to Holt, the two conceived a scheme for automating the process. A schematic of their preliminary design is included (see Figure 1, Appendix A). This preliminary design served as a guideline and basis from which to construct the working model now being tested. The actual schematic of this model is shown in Figure 2, Appendix A with modifications and deviations from the original conception shown in red.

### 2.0 PROBLEM AND RESULTS

### 2.1 PROBLEM

Operation of the total carbon system (TCS) to run a typical batch of $50-80$ samples requires the undivided attention of a technician for up to five hours.

After properly calibrating the machine, the technician must perform the following steps for each sample. (see Table I)

## Table I

Steps Required for the Manual Operation of TCS

1. Turn on zero gas.
2. Put valve in bypass position.
3. Put adapter on vial.
4. Place vial in position.
5. Screw vise up.
6. Acquire adequate seal (check).
7. Turn on flow valve.
8. Purge for ten seconds.
9. Put valve in analyze position.
10. Turn off flow valve.
11. Pull down on jaw and turn. (Break vial.)
12. Raise jaw back up.
13. Push purge tube down to within one inch of bottom of ampule.
14. Turn on flow valve.
15. Lower purge tube to within $1 / 8$ inch of the ampule.
16. Check flow rate (Continuous).
17. Wait for needle to peak and return to $5 \%$.
18. Switch to bypass.
19. Wait for integrator to stop.
20. Push "PRINT" button.
21. Turn flow off.
22. Drop vise.
23. Raise purge tube and jaw apparatus.
24. Remove ampule.

It is desired to automate this process. This would free the technician, enabling him to perform other tasks in the laboratory.

### 2.2 RESULTS

A working model has been built to relieve the technician of the necessity of his constant presence at the machine. With the TCS properly calibrated and the automation system properly interphased, the technician need only perform the following steps:

## Table II

Steps Required for Start-Up of TCS Automation

1. Load ampules into casings (inspecting each o-ring).
2. Load ampules and casings into gravity feed rack.
3. Turn system on.

The flow rate will tend to decrease durind a "run" (scc IMPROVEMENTS, PURGE mUBE) and must be periodically adjusted. Rather than automate this, an alarm is providea to alert a nearby technician of the necessary adjustment.

The working model existing is not "state of the art.". It has been built after a preliminary design which was modified several times in the design/construction process. This model can be used to evaluate (see EVALUATION PLAN) the automation system and perform any adjustments or modifications toward producing a marketable apparatus.

### 3.1 PROBLEM

As indicated in PROBLEM, the operation of the TCS depends on the methodical completion of a series of steps. These steps must be completed in order and with careful attention given to nonitoring the flow iate. Adequate time delays must be allowed at certain points to ensure time to purge all atmospheric impurities and to allow sufficient time to qualitatively take each sample.

### 3.2 ALTERNATIVES

In keeping with the design objective of running with minimum supervision and possibility of destroying samples, the alternative of staggering each function with pre-set time delay was rejected. The amount of time needed to measure each sample varies directly with the level in each ampule. With less seawater in the ampule, more $\mathrm{CO}_{2}$ is able to come out of solution prior to sampling, thus less sampling time is required to bubble dissolved $\mathrm{CO}_{2}$ out of the sample and the measuring process begins and is concluded sooner. A distinct disadvantage of depending solely on time delays for initiating each function is the possibility of the device continuing to run through its procedures regardless of a jammed ampule and possibly destroying some of these samples.

The implementation of "limit switches" placed strategically to sense the completion of each function and to activate the next step would forego the possibility of a "run-away" machine.

### 3.3 SELECTED DESIGN

The actual design employs both the limit switch array and time delay relays. The time delay relays serve different functions.
"TIME DELAY RELAY I" (see Figure 2. App.A) allows the needle sufficient time to pass the $5 \%$ reading on the LIRA metex initially so the sensor will detect the needle only on its way back down. This observation was formerly made by the technician.
"TIME DELAY RELAY 2" provides time for both BYPASS and FLOW valves (deactivated by "PURGE" relay) to flush the "LIRA" analyzer of residual sample gas. This causes the needle of the "LIRA" meter to drop rapidly from "5\%" to "0." The length of time required for the needle to span this distance without purging was deemed too long. This method of purging the last $5 \%$ was employed in the manual operation of the TCS and automating this procedure should improve accuracy of data.
"TIME DELAY RELAY 3" allows up to 10 seconds for purging the cap/ampule annulus of atmospheric gas. (See SAMPLE, SELEECTED DESIGN)
"TIME DELAY RELAY 4" requires that the ball of the ROTOMETER remain at 13 (the desired position) for at least l-2 seconds. This prevents misinterpreting the ball merely "passing by" as stabilized flow. Note that if this flow is NOT correctly stabilized, an alarm is activated to alert the technician that an adjustment must be made.

### 4.0 FEED DISPOSAL

## 4.Al PROBLEM

To convey each ampule, individually, to the sampling position, then to stop each ampule precisely at this point consistently and pause at this position until sampling has been completed.

## 4.A2 SELECTED DESIGN

An indexing system was designed and is depicted in Figure 1, Appendix B.

The indexing wheel has a radius of curvature which, upon rotation, tends to guide the next ampule into its respective socket. With each $90^{\circ}$ rotation of this wheel, three ampules are moved. (Refer to Figure 1, Appendix B.)
"Ampule 1 " has been measured and thus is discarded.
"Ampule 2 " is relocated at the sampling position replacing vial l. A hinged, spring-loaded plexiglass door serves as a retainer, preventing toppling of the ampule or over-travel due to the incurred momentum. It was found that a "doorstop" was necessary to prevent the ampule's casing (see SEAL) from being forced against the indexing wheel thus inpeding the sealing process.
"Ampule 3 " ropJaces "ampule 2 " at the "ready" position.
4.A2.l DRIVE MECHANISM

The indexing wheel is driven by the mechanism shown in Plate l, Appendix E. (Refer to Plate 1, Appendix B):

Upon completion of the series circuit of limit switches LS-1, LS-3, LS-5, (Figure 2, Appendix A), $\mathrm{N}_{2}$ activated cylinder CL-I extends. The dowel-pin in Piece A catches the teeth of Gear $B$ thus rotating it. CL-1 is mounted on a pivot, allowing A to follow the curvature of Gear B. Gear B and Gear D are affixed to the shaft by means of a woodruff key (as is the indexing wheel). Mounted opposite Piece A is a pawl. When Piece A has rotated the indexing wheel to the point where an ampule is in precisely the measuring position, Piece A pushes the pivot-mounted pawl into the buttress teeth of Gear D. Cylinder CL-l has not yet reached its maximum stroke, thus locking the ampule into exactly the correct position. Note that the travel of Piece A from its original position to this one opens limit switch LS-l and closes LS-2. The mechanism will remain locked in this position until the measuring and metering of this sample is completed. After completion of this measuring, the piston of cl-l retracts, its springloaded mounting returns it to its original position as does the spring-loaded pivot of the pawl. Notice that this reopens LS-2 and closes LS-l which will start the process over again (Figure 2, Appendix A).
4.B1 PROBLEM (Continued)

A means for delivering each consecutive ampule to the "ready" position of Ampule 3 (Figure 1, Appendix B) is still required. The "feed" characteristic of the index wheel's curvature relies on the assumption that the feeding ampule is
constantly pressed up against the indexing wheel. This function must also be provided.
4.B2 ALTERNATIVES

## 4.B2.1 GRAVITY FEED

This would be a kind of chute or ramp which the ampules slide down to the "ready" position. This would require the ampules going from an incline-induced angle to the upright position. The slight angle necessary to deliver the ampules upright at the "ready" position would not provide the ampules enough momentum to exert sufficient pressure against the indexing wheel to be "picked up."

## 4.B2.2 CONVEYOR BELT

A conveyor belt could be used to carry ampules to the "ready" position. It could be powered externally (e.g., an electric motor) or conceivably by the indexing shaft, although this would involve relatively complex gearing or pulley adaptations. It would not be practical to provide a belt long enough to accommodate up to 80 ampules, but this system could be incorporated with a gravity feed system.

## 4.B2.3 CONVEYOR DISC

This is essentially the same concept as the conveyor belt, but more easily adapted to the indexing shaft.

A hybrid system, combining both the gravity feed and the conveyor disc was employed (see Figures 2 and 3, Appendix B). The gravity feed ramp shown and currently being used is not "state of the art" but merely a temporary model being used to determine critical angles, loading techríques, etc.

The retaining fences are designed such that the ampule coming off the gravity ramp is tilted to land upright on the conveyor disc. These fences guide the ampule to the "ready" position and prohibit toppling. The innermost fence continues to guide the ampule en route to the measuring position.

The conveyor disc is mounted on a $1 / 4$-inch shaft. On the other end of the shaft is mounted a grooved pulley. A similar pulley is mounted to the base of the indexing shaft. The two pulieys are connected by an o-ring twisted $130^{\circ}$. This twist causes the conveyor disc to rotate in the opposite direction of the indexing wheel, thus feeding the ampules up against it. Through several iterations, it was found that a pulley ratio of $2: 1$ both fed ampules at a sufficient rate and ensured constant pressure of the next ampule against the indexing wheel.

### 5.0 SEAL

### 5.1 PROBLEM

It is necessary to create a seal around each ampule to be sampled. This is to allow the trapped air to be purged and to preclude intrusion of any impurities after sampling has begun. Formerly, a technician would place a cap and gumrubber seal on each ampule and force it into a receptacle by screwing a vise-like pedestal up against the ampule's base.

### 5.1.1 ALTERNATIVES

5.1.1.1 One Sealing Cap Fixed to System

This would seemingly be the most simplistic and economical solution: one scaling cap, equipped with an O-ring which would lower upon each ampule in the measuring position. At the top of the cap would be the glass-breaking/sample-taking apparatus, also sealed against the atmosphere. This, however, would require that a seal be made for each sample. Since the sampling process involves breaking the ampules, some glass from the last ampule would inevitably be left in the cap. This broken glass would, of course, accumulate and eventually hamper the proper sealing of the sample and cause damage to the o-ring. A damaged o-ring would not be discovered until the end of a "run" of samples, thus resulting in several ruined samples.

### 5.1.1.2 Individual Casings

If properly designed, individual casings, one for each ampule, would retain and carry away the broken glass of each sampling. The O-ring in each casing would be visually inspected before inserting each ampule, thus ensuring a proper seal. These casings would also serve to protect the ampules during the FEED process.
5.2 SELECTED DESIGN

For those advantages stated above, the individual casing method was employed. The nature of this design requires that the caps be open at the top to allow the glass-breaking/ measuring process. This creates a need for another seal, one between the top of the cap and a receptacle housing the glassbreaking/measuring apparatus (the process chamber). (See Figues 1 and 2, Appendix C).

The automated seal-making process is much the same as the manual operation. Once the vial has been indexed to the "measuring position" subsequently closing LS-2 (see Figure 1, Appendix B) the circuit activating cylinder CL-2 (Figure 2 , Appendix $A$ ) is activated. Attached to this cylinder is. Pedestal E (Plate 1, Appendix B) which is directly centered and flush with the "measuring position." The Process Chamber $F$ (Plate 2, Appendix C) is directly above this pedestal. Thus, completion of the LS-2 circuit activates CL-2, driving the capped ampule into the process chamber. As with CL-1, the
piston of CL-2 does not reach the end of its stroke, thus ensuring a tight seal against the gasket (Figure 2, Appendix C). At the top of its travel, (ampule sealed in the process chamber) Pedestal E closes LS-4. LS-3 is open in this position. (Refer to Figure 2, Appendix A).

NOTE: At this point LS-1 and LS-3 are open, LS-2 and LS-4 are closed. The closing of LS-4 activates the circuit allowing a time delay for flow rate verification (see TIMING).

### 6.0 SAMPLE

### 6.1 PROBLEM

After achieving a proper seal, the air trapped in the process chamber must be purged with $N_{2}$. The top of the ampule must then be broken off and a tube lowered into the seawater sample to within $1 / 8$-inch of the bottom of the capsule. $N_{2}$ is to pass down through this tube to bubble up through the sample, bringing dissolved $\mathrm{CO}_{2}$ out of the seawater. This mixture of $\mathrm{N}_{2}$ and sample gas must be collected and sent on to be measured.

### 6.1.1 ALTERNATIVES

6.1.1.l Separate Glass-Breaking Jaw and Sample Tube

This is the method employed in the original (manual) operation. A heavy, toothed tube is lowered and twisted to break the ampule. Down through this tube, another smaller tuve is lowered into the sample. $N_{2}$ is bubbled through the sample from the smaller tube and the sample gas is carried up through the annulus of the larger crushing tube. The poor design of this system causes tedious manipulation on the part of the technician. (See PROBLEM, steps ll-15).

### 6.1.1.2 Combination Breaking Jaw and Sample Tube

It was determined that a better design would be to use a larger sample tube. Such a tube could bubble the sample more vigorously from a further distance from the ampule's bottom.

This would prevent cavatation and lessen the possibility of carrying water droplets along with the sample. This stouter tube would not only forego the raising and lowering of the sample tube (PROBLEM, steps 11-15 to prevent cavatation) but could also be used as the glass breaker as well.

### 6.2 SELECTED DESIGN

Through destructive testing, it was found that 3/16-inch stainless steel tubing with small notches cut in the end worked with a $100 \%$ success rate in smashing through the tops of the ampules without shattering the shoulder where the seal is made (see Figure 1, Appendix C). This notched tube is threaded into Manifold $H$ (Plate 2, Appendix C) and passes through the Gland G. This tube passes through two o-rings (Figure 2, Appendix C). This not only provides a back-up system (one o-ring can be damaged during a "run" without ruining all the samples), but also aids in ensuring proper alignment.

Upon achieving a proper seal, $\mathrm{N}_{2}$ is constantly passing through the annulus formed by the casing and ampule. This purges the trapped atmospheric air prior to crushing the ampule.

Time Delay Relay 3 (Figure 2, Appendix A), activated by the closure of LS-4 allows up to ten seconds for the flow rate to stabilize. If the flow rate has not stabilized by the end of ten seconds, the alarm sounds alerting the technician and halting the entire process until the flow rate is corrected. If, however, the flow rate is correct at the end
of the provided time delay, the circuit which activates CL-3 is energized. This sends the sample tube (fixed to CL-3 via Manifold H [Plate 2, Appendix C]) down, breaking through the top of the ampule. At this point, Manifold $H$ is no longer holding closed LS-5, but is now holding closed LS-6.

### 7.0 INTERPHASE

### 7.1 PROBLEM

In order to prevent erroneous data, the LIRA infra-red analyzer must remain purged with $\mathrm{N}_{2}$ between samples. The sample gas being carried to the analyzer by the $N_{2}$ must be scrubbed free of moisture. Once the sample reaches the infrared analyzer, the $\mathrm{CO}_{2}$ content is indicated by registering on the meter. The needle of this meter will peak at some point and then slowly begin to fall. As the needle registers, an integrator is activated. Integration continues until the needle reaches zero. Once the integrator stops (needle at zero) the technician pushes the "PRINT" button to record the data on a printer. Flow rate of the sample gas must also be constantly monitored.
7.AI SENSING CRITICAL READINGS

## 7.A2 ALTERNATIVES

7.A2.1 Replace Existing Meters

Meters are available "off the shelf" with adjustable sensors. These could be used to replace the flow meter and the LIRA meter on the infra-red analyzer and adjusted to detect the critical readings. This would not be in keeping with the design objective to avoid altering the existing system as much as possible.

In order to ensure the gas reaching the infra-red analyzer is dry and meets the maker's specifications, it is actually run through the TCS drying system.

Some slight modifications have been made on the routing of sample gas through the TCS, as shown in Figures 2-5, Appendix D. (For detailed discussion of this, see IMPROVEMENTS MADE ON EXISTING TCS).

## 7.Bl.1 Routing of Sample Gas

As stated previously, with the sample tube down in the "sample" position, LS-6 is closed.

NOTE: At this point, LS-1, LS-3, LS-5 are open. LS-2, LS-4, LS-6 are closed.

The closure of LS-6 activates the following:

1. Time Delay Relay 1 , which allows MSA needle to pass 5\% on its way "up" (TIMING).
2. Diverts the "bypass" valve from simply passing $N_{2}$ through the flow meter (to be adjusted) and then exhausting it, to directing it to the "flow" valve.
3. Diverts the "flow" valve from merely running pure $\mathrm{N}_{2}$ through the infra-red analyzer (to kẹep it purged), to passing the sample gas (from "bypass") on to the meter for reading.

In the design and interphase of the TCS automation system, certain manufacturers' designs were modified for the automation which also improved the existing system:

## $8.1 \mathrm{~N}_{2}$ VENTING

As seen in Figure 4, Appendix D, the entire time a sample is being taken by the $T C S$, unused $N_{2}$ is being vented out of the machine. This amounts to substantial waste. The re-routed TCS automated system (Figure 6, Appendix D) does not employ this wasteful feature.

### 8.2 PURGE TUBE

The combination glass-breaking/sample tube (see SAMPLE), together with the large volume created between the process chamber and the ampule cap (see Figure 2, Appendix C) reduces the amount of water that can accompany the sample gas. This reduces the frequency with which the $\mathrm{Mg}\left(\mathrm{CLO}_{4}\right)_{2}$ in the drying tubes must be changed. With the extended life of. the $\mathrm{Mg}\left(\mathrm{CLO}_{4}\right)_{2}$, flow restriction is lessened and therefore the flow rate need not be adjusted as often.
8. 3 5\% PURGE

Installation of the R-F PROXIMITY switch ensures that the final purge (from $5 \%$ to 0 -- see TIMING, SELECTED DESIGN) occurs at precisely the same point for each sample. This was formerly estimated (visually) by a technician. This mechanization must improve accuracy in data gathering.

### 9.1 CRUSHING CYLINDER BORE

CL-3 (Plate 2 , Appendix C) should be replaced with a cylinder with a bigger bore. This would enable the cylinder to exert more force to break ampules at a lower operating pressure.
9.2 FEFD CYLINDER RELOCATION

With a modified design, CL-l (Plate 1, Appendix B) could be relocated in a less obstrusive manner.
9.3 TIME DEIAY 1 FOR R-F PROXIMITY SWITCH

Although this function works on the existing model, it does so by a very slight margin. Ten seconds is allotted for the needle of the IR analyzer to initially pass the $5 \%$ reading. The samples tested thus far have come very close to this limiting valve and it is conceivable that a sample could exceed it. This would cause an incorrect reading of that sample.

The simplest means of correcting this potential malfunction would be to move the $R-F$ sensor further down-scale (e.g., 2-3\%). As before, if all samples are taken at precisely this point, no sacrifice to accuracy should be incurred.

This piece is presently made of delrin. Due to the force exerted on this piece by the ampule crushing procedure, it is suggested that another similar one be made of stainless steel. This would tend to reduce the stripping of the threads where the purgn tube is inserted which is a critical seal.

## 10. EVALUATION PLAN

The evaluation plan is provided to determine whether any sacrifice has been made in implementing the TCS automation system.

Place each of twenty-five different samples into two separate ampules (thus yielding fifty ampules of identical pairs). These are to be identified (e.g., 1A, 1B--2A, 2B, etc.). One set (e.g., $1 A-25 A$ ) is to be sampled in order manually on the TCS. The remaining set (in this case, 1B-25B) is to be loaded in the same order in the TCS automation system. The data gathered from these runs should be analyzed. to determine if any significant statistical deviation occurs.

## 11. EXPENDITURES

| Quantity | Description and Mfg. Part No | Each ${ }^{\text {P }}$ | Total |
| :---: | :---: | :---: | :---: |
|  | Unimax Snap-Action Switches |  |  |
| 2 | 2TMT18-4 \#07F033 | \$1.15 | \$2,30 |
| 4 | 2TMA15-4 \#07F031 | 1.35 | 5.40 |
| 2 | 5LM-E \#07F061 | 2.20 | 4.40 |
| 2 | 2LML-E \#07F062 | 2.10 | 4.20 |
| 2 | 2LMW-E \#07F060 | 2.45 | 4.90 |
| 5 | Sigma 77R2-120AC Relays \#38F1669 | $4.30$ | 21.50 |
| 6 | Sockets for Above \#38F1608 | 1.20 | 7.20 |
| 4 | Cramer 3800 Solid State Time Delay Relays \#3822 | 25.50 | 1.02 .00 |
| 4 | Octal Sockets Bunker Ramo 78S8 \#38F816 | . 35 | 1.40 |
| 1 | ```Mallory Sonalert SCllo #64F303``` | 9.92 | 9.92 |
| 6 | MAC Solenoid Valves \#224-111B | 23.00 | 138.00 |
|  | Bimba Air Cylinders |  |  |
| 1 | \#024-D | 10.45 | 10.45 |
| 1 | \#061.5 | 12.45 | 12.45 |
| 1 | \#024 | 12.45 | 12.45 |
| 3 | Brackets for above | . 95 | 2.85 |
| * | Assorted Fittings (See Requisition \#33140) |  | 152.50 |Assorted Fittings (See152.50

## Quantity

| 1 | Photocell Transmitter/ <br> Receiver <br> EP120-14660/EP120-14560 | 69.00 | 69.00 |
| :--- | :--- | :--- | :--- |
| 1 | Photocell Amp EP150-10.000 | 95.00 | 95.00 |
| 1 | R-F Sensor EE951-263000 | 30.50 | 30.50 |
| 1 | Proximity Switch <br> EE971-02103 | 166.50 | 166.50 |
| 1 | Socket for Above <br> EE012-5004 | 10.00 | 10.00 |

## 12. ACKNOWLEDGMENTS

To John K. Holt: for his guidance, infinite knowledge, and his wizardry for making things work

To John Montgomery and Gary Peterson: for use of their laboratory and an unlimited supply of ampules and humor

To Bruno Petersen and Mike Hyde: for guidance and valuable time in the machine shop

To Roy Spor: for advice and much patience

APPENDIX $A$

General


APPENDIX B

Feed

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## APPENDIX C

Seal/Sample

FIG. 1



PLATE 2

## APPENDIX D

## Interphase







