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**AN AUTOMATIC MONITOR FOR As^{76} CONCENTRATIONS
IN REACTOR COOLING WATER**

H. G. RIECK

APRIL 1963

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AN AUTOMATIC MONITOR FOR As^{76} CONCENTRATIONS
IN REACTOR COOLING WATER

By
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Chemical Research
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April 1963

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AN AUTOMATIC MONITOR FOR As^{76} CONCENTRATIONS IN REACTOR COOLING WATER

INTRODUCTION

The purpose of this study was to provide a prototype instrument to monitor the As^{76} concentration in reactor cooling water streams. This report describes the development, design, and operation of the prototype monitor.

As^{76} is one of the biologically significant radionuclides which is released to the Columbia River in Hanford reactor effluent water in very dilute concentrations. While the concentration of As^{76} is far below dangerous pollution levels, it is still desirable to develop automatic techniques for monitoring the amounts that are discharged to the river. Short term fluctuations and general trends in the concentration would be available without manual sampling and analysis, and a historical record of the As^{76} concentration would be provided.

SUMMARY

A system was developed for monitoring As^{76} concentrations in reactor cooling water. Gamma energy measurements are made on a sample which has been pretreated with cation exchange resin and decayed for 3 hr to reduce interference. Additional correction for isotope interference is applied during the counting step.

A prototype monitor for As^{76} was fabricated and tested. A sample is analyzed every hour and the result is recorded in digital form. The minimum detection limit is 20 picocuries of As^{76} per milliliter of reactor cooling water. The monitor has provided reliable operation with a minimum of service requirements.

SYSTEMS DESIGN

The main objective of the study was to develop a monitoring system which required a minimum of maintenance and attention, yet which would provide reliable detection of a low As^{76} concentration in reactor effluent

water. Since the monitor might be used at various locations during production tests, the design requirements included portability.

As⁷⁶ in reactor water can be isolated for measurement by several methods. Extraction of As⁷⁶ can be made into chloroform from a butanol, ethyl acetate, water mixture.¹ This method requires stepwise addition of chemicals and the separation of a water-organic phase. In a second method, As⁷⁶ is separated from interfering isotopes by distilling from a HBr solution as the bromide.² The distillation requires pretreatment with HClO₄ and H₂O₂ and a reduction in volume by evaporation. The solubility of arsenic chloride in benzene is the basis of another analytical technique.³ This method requires volume reduction and addition of HCl followed by extraction into benzene.

The use of any chemical separation method in a continuous automatic monitor would require automation of several steps. A complex mechanical system would be necessary and would result in problems of operational reliability, high fabrication costs, and excessive maintenance requirements. A simple chemical removal of a bulk of the interfering isotopes followed by an electronic correction in the detection step would be the simplest system. A previous method⁴ used in another automatic monitor was ion exchange. The exchanger removed the major interfering cations such as Zn⁶⁵, Cu⁶⁴, Na²⁴, and Mn⁵⁶. The As⁷⁶ concentration was then determined in a dried sample by a gamma energy measurement at 0.56 Mev.

The cation exchange resin technique offered the most desirable system to automate;⁵ the sampling system could be a resin column and a sample cell located over a scintillation crystal. The presence of Cl³⁸ caused an interference problem, and chemical removal without drastically affecting the As⁷⁶ was not successful. The short half-life of Cl³⁸ (37 min) suggested that a batch sampling system be used rather than a continuous flow to provide the means for obtaining a holdup time for decay purposes.

Since the concentration of As^{76} was not expected to fluctuate rapidly, a sampling frequency of 1 hr appeared adequate.

A digital counting system was proposed because:

- Low concentrations of As^{76} were anticipated and a digital system would allow an accumulation of counts for a finite counting interval.
- Considerable variations in background might occur at the monitor location. A background correction could be made for each sample with a digital system.
- Interference from other isotopes was anticipated in the 0.56 Mev As^{76} channel. A subtraction count determined by a ratio of counting times would correct for the interferences. A digital system could provide an accurate method for making this correction.

MONITOR DESCRIPTION

The As^{76} monitor is enclosed in an 84 in. tall, 22 in. wide, and 22 in. deep instrument rack which contains the electronic circuitry, sampling system, and the shield-detector assembly. The monitor is shown in Figure 1. The assembly weighs about 700 lb and is equipped with casters. The electronic circuitry is mounted on the front panel and consists of a programmer and a single channel pulse height analyzer equipped with a scaler and printer.

The sampling system, which is readily accessible from the rear, consists of a pump, a cation exchange resin column, four sample reservoirs, and a sample cell.

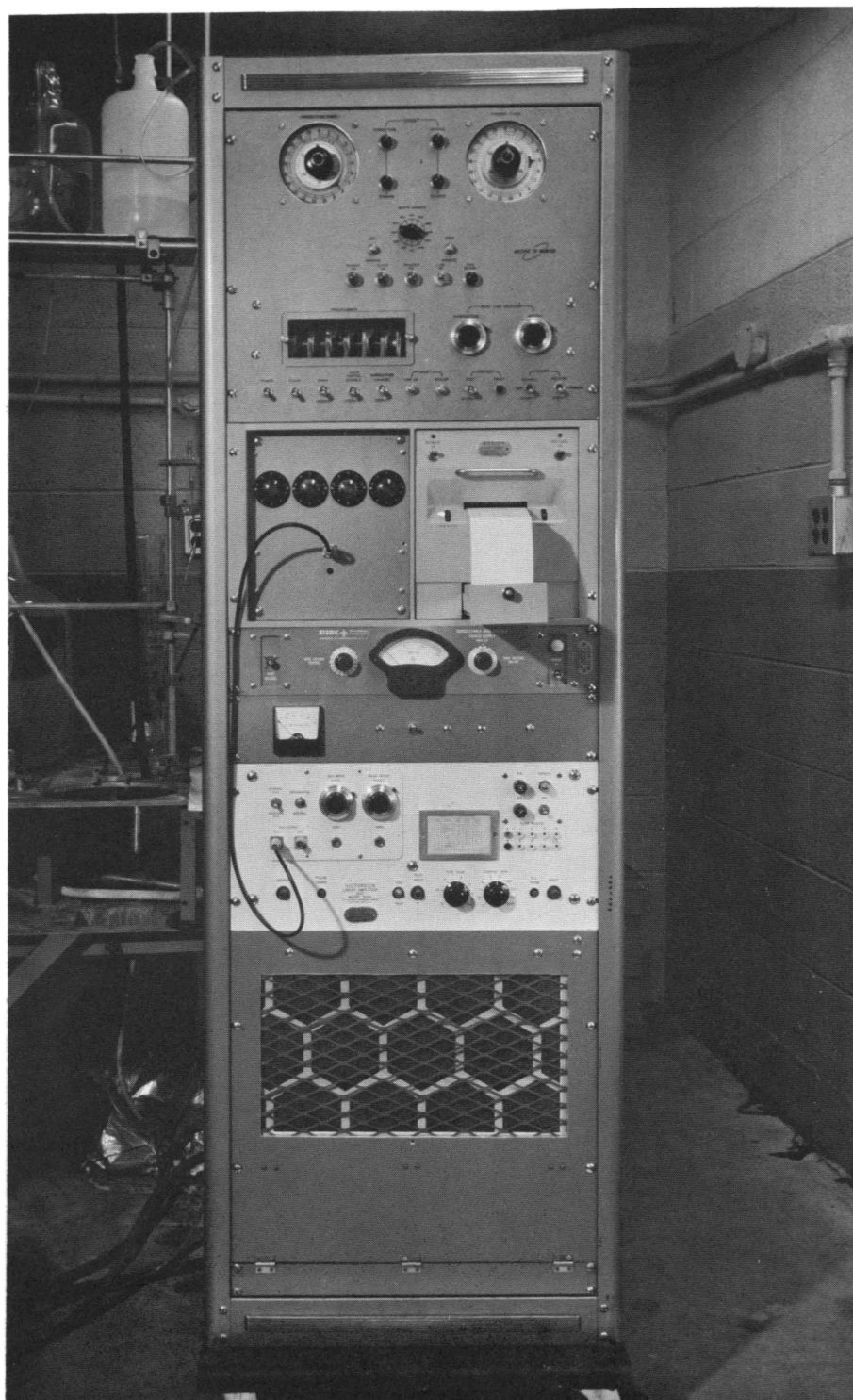


FIGURE 1

As⁷⁶ Monitor

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Sampling System

The reactor effluent water is pumped to a glass column containing 500 ml of cation exchange resin (200 mesh). The sample flow rate of 300 ml/hr is controlled by the pump. Downward flow through the column is maintained by a variable head of sample above the resin. As back pressure across the column increases, the head over the resin automatically increases and the flow is maintained at the amount delivered by the pump. Overflow to the drain is provided if the back pressure becomes excessive.

The column effluent which is depleted of cation isotopes such as Na^{24} , Zn^{65} , Cu^{64} , and Mn^{56} , is directed to one of four reservoirs. The glass reservoirs contain 275 ml of sample. Excess sample overflows to a drain line to insure that a full volume is delivered each time. Collection time for each sample is 1 hr.

Two hours later, the sample is released to the sample cell. After 30 min, two gamma counts are taken and the sample is drained from the cell. Flow through the entire system is controlled by programmer actuated solenoid valves. A flow diagram of the monitor is shown in Figure 2.

Glass components are used, where possible, to permit visual observation of the sampling system operation. Plastic tubing is used to interconnect the various components. The resin column, sample reservoirs, and the solenoid operated control valves are mounted on the rear door as shown in Figure 3.

The 275 cc sample cell is fabricated from glass and is equipped with an overflow and a drain port. The cell is located above the crystal detector and the entire assembly is enclosed in a 3 in. thick lead shield. The shield, detector, and sample cell are shown in Figure 4. The shield, assembled from 3 x 4 x 8 in. lead bricks, should be removed if the monitor is moved.

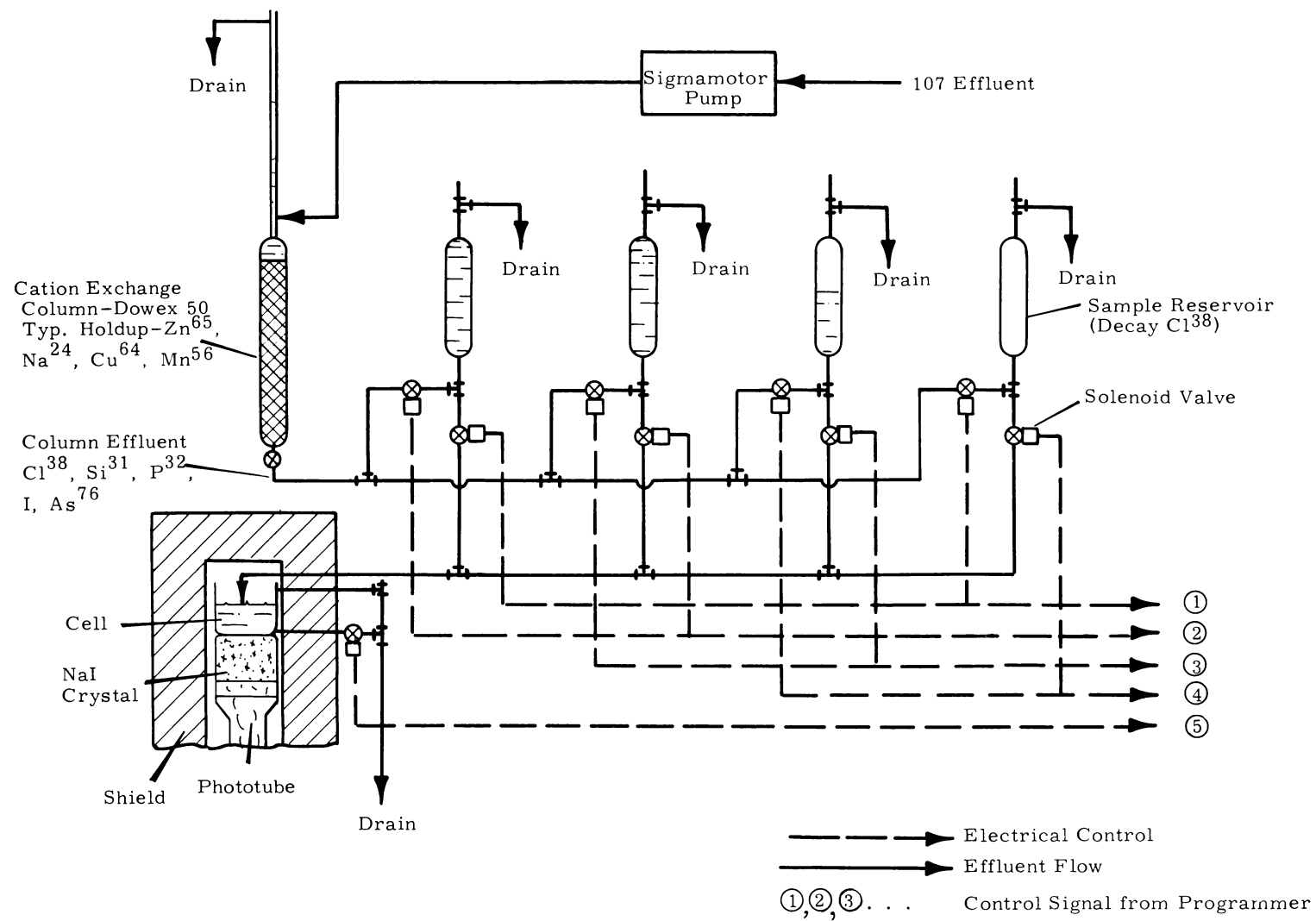


FIGURE 2
 As^{76} Monitor Sampling System

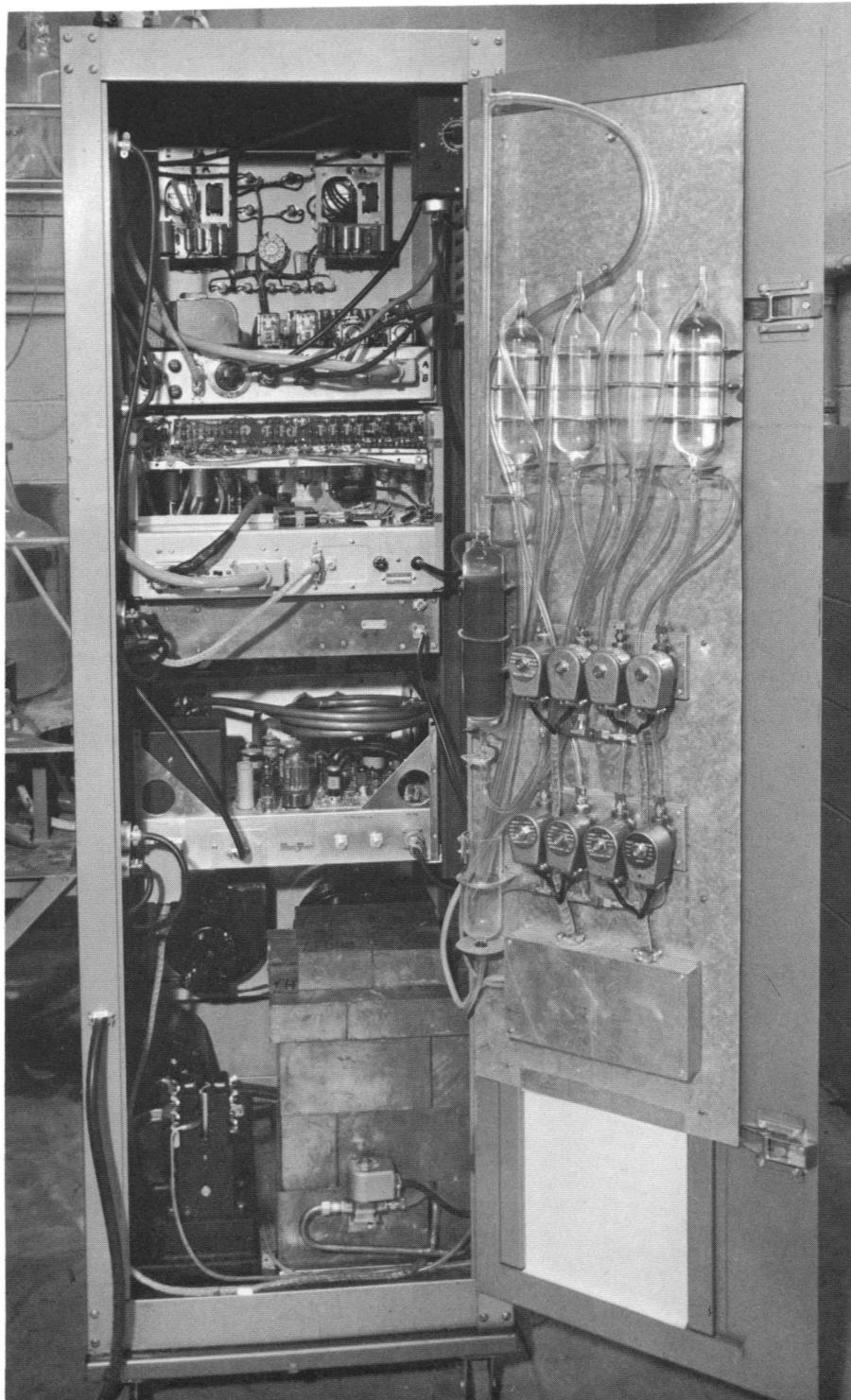


FIGURE 3

As⁷⁶ Monitor

View of Sampling Equipment, Shield, and Electronics

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FIGURE 4

Detector, Shield, and Sample Cell

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Electronics

The As^{76} activity in the sample is measured with a single channel gamma ray spectrometer system. A block diagram of the counting system is shown in Figure 5.

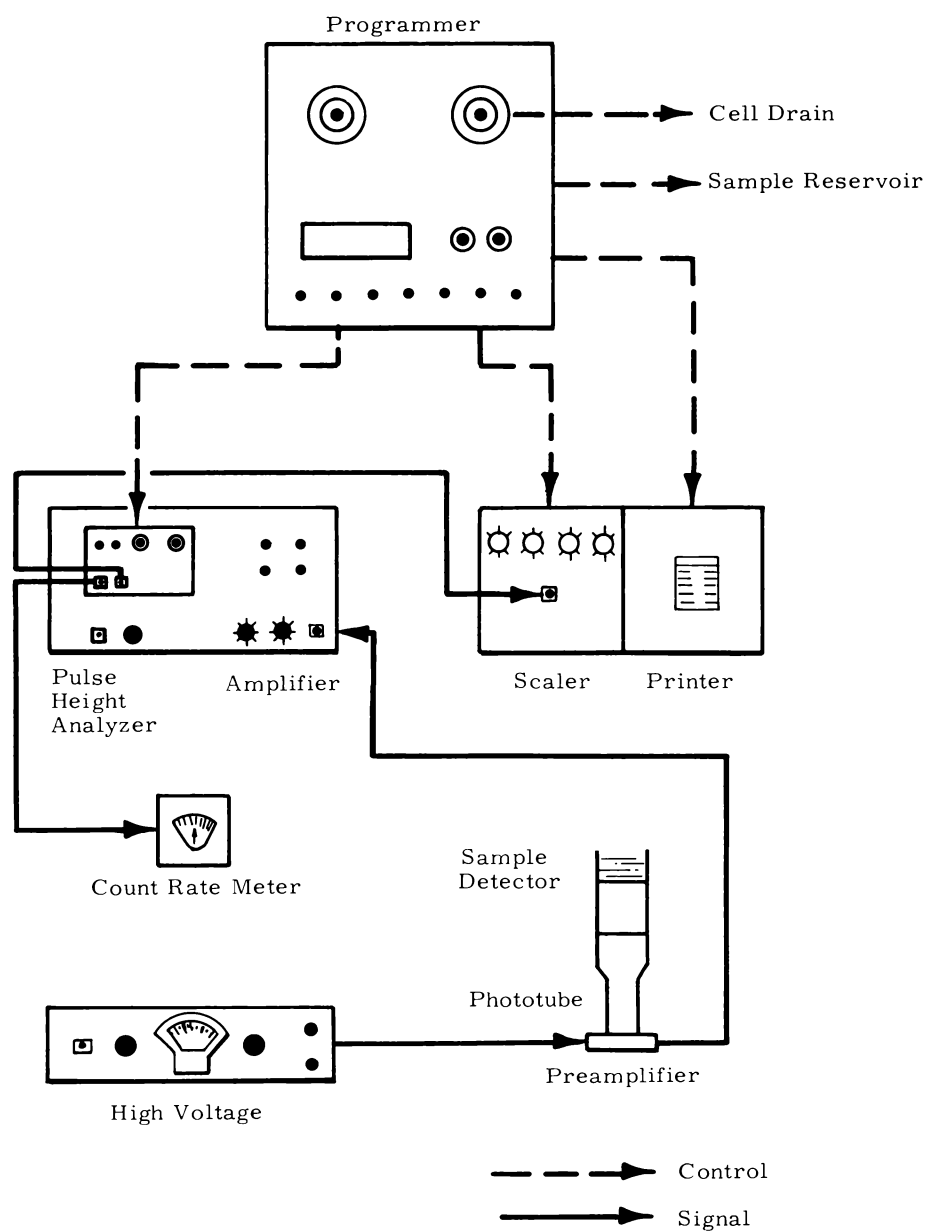


FIGURE 5

Electronic System of As^{76} Monitor

The gamma photons from the sample are detected by a 3 x 3 in. sodium iodide (thallium) crystal mounted on a 3 in. multiplier phototube. A transistorized preamplifier, located at the base of the phototube, feeds the signal to a linear amplifier. The amplifier gain is set so that the 0.661 Mev gamma photopeak from Cs^{137} occurs at 33 v on the pulse height analyzer when the analyzer window is adjusted to 10 v. A transistorized count rate meter is provided to assist in making gain adjustments of the amplifier. Counts from the pulse height analyzer are accumulated on a decade scaler which is located in the left half of a digital printer. Data from the scaler are recorded in digital form by the printer. A programmer controls the sample handling, counting, and data recording functions.

Commercially available instruments are used where possible with the exception of the scaler and programmer. These units were designed and built to fulfill requirements which were unique to this system. A brief description of the two instruments follows.

Scaler

The four decade scaler is located in the left side of the printer unit. The components are mounted on a front panel which can be tilted forward for servicing access as shown in Figure 6. Glow transfer tubes which allow forward and backward counting are used as scaling stages. The tubes are equipped with a base connection from each of ten cathodes to provide a signal for data recording purposes. A driver circuit for each decade is composed of four 2N398 transistors; two negative pulses of necessary timing and duration are generated for each input pulse and transmitted to the glow transfer tube by an emitter-follower circuit. The scaler circuit details are in Figure D-1 in Appendix D. A description of the driver circuit operation is included in Appendix A.

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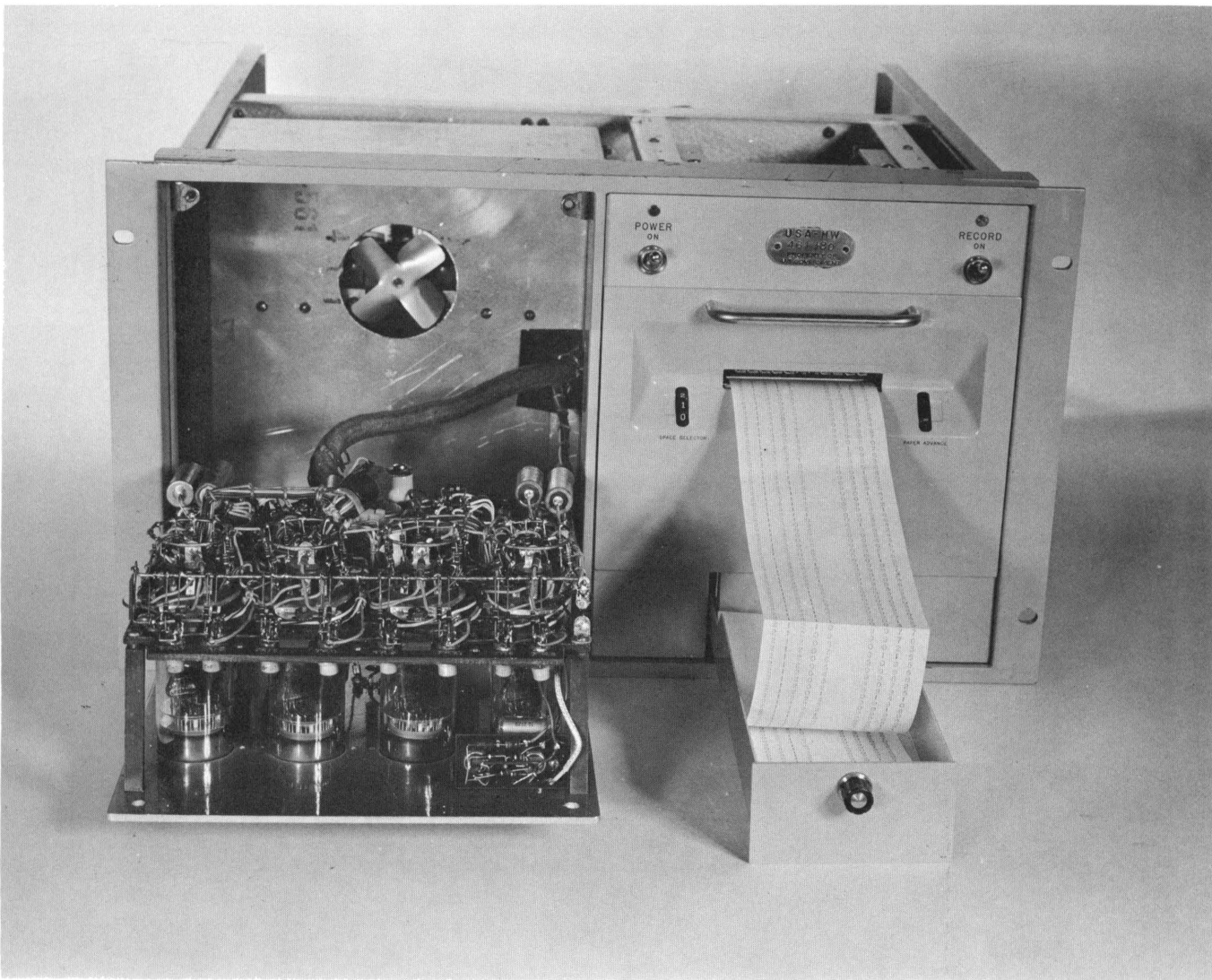


FIGURE 6
Scaler and Printer Chassis Showing Scaler in Position for Servicing

Programmer

Circuits which control counting intervals, data recording, and sampling functions are located in the programmer. A 1 revolution/hr timer is used as the basic sequence timer. Two additional timers measure the counting intervals on signal from the sequence timer. Switches, which are located on the front panel, bypass the programmed sequence and are used during routine maintenance and service operation.

The programmer controls:

- The resetting of the scaler before data accumulation.
- The backward counting to correct for background and isotope interference.
- The switching of the pulse height analyzer base line at the proper time for accumulation of an isotope interference correction.
- The draining of the sample cell so that a background subtraction count can be accumulated.
- The energizing of the solenoid operated valves to permit collection, holdup, and release of the sample to the counting cell.
- The printing of accumulated data.
- The providing of time and date coding information to the printer for sample data identification purposes.

Power supply circuits for the glow transfer, transistor circuits, and solenoid valves are located on the programmer unit. A "check" circuit requires that switches be set in proper position following a routine maintenance or servicing procedure.

The programmer circuit details are in Figure C-1 of Appendix C along with a written description of the programmer sequence.

Operation

The sample is collected at the rate of 5 cc/min for 1 hr. The sample is stored in one of four 275 cc reservoirs equipped with overflows to drain excess liquid. Four reservoirs are used to provide a 3 hr delay

before counting so that interference from 37 min Cl^{38} is negligible. At the beginning of an hour cycle time, the programmer causes the solution in a reservoir to drain to the sample cell for counting. The reservoir which contained the preceding sample is switched to the resin column outlet and is filled during the next hour.

After about 30 min, the programmer signals the start of a gamma count at 0.56 Mev. The counts are accumulated on the decade scaler as a forward count. After 5 min of counting time, the programmer signals for a backward count at 0.8 Mev for 8 min. This count corrects for interfering radioiodine. At the end of this period, the programmer signals the sample to be drained and a background count in the As^{76} channel is accumulated on the scaler in the backward direction. Following this count, a background accumulation is made in the correction channel in the forward direction. At the end of this period, the counts which have been accumulated on the scaler during the counting interval are recorded in digital form with the printer. The data and counting time are also printed. A sample of the printed data is shown in Table I.

TABLE I

As^{76} MONITOR DATA OUTPUT

	9 16 12	0621
	9 16 13	0427
	9 16 14	0451
	.	.
	.	.
	.	.
	9 16 23	0577
	9 17 00	0616
	9 17 01	0492
Month*—	Day	Hour
		Counts/sample (picocuries/ml = counts x 0.02)

*Months: January through September—1 through 9
October—0
November—
December—blank

DISCUSSION

Isotope Interference

The halogen isotope anions pass through the cation exchange resin column and interfere with the As^{76} measurement. Compton interference errors result due to Cl^{38} . A gamma spectrum of the cation exchange column effluent is shown in Figure 7. The photo peak at 2.2 Mev is from the Cl^{38} decay and a Compton interference at 0.56 Mev causes a 15% error in the As^{76} measurement. At the start of the counting period, 3 hr later, the Cl^{38} has decayed sufficiently so that the error is less than 1%. The spectrum at the start of the counting period also is shown in Figure 7.

Various radioiodine isotopes are present and contribute an error in the As^{76} analysis. The error is due to Compton interference from decay of I^{135} , I^{133} , and Xe^{135} (daughter of I^{134}). The decay of I^{135} causes an error due to the 0.51 Mev gamma. The gamma spectrum of the radioiodine in the cation column effluent, obtained by making a CCl_4 extraction on the monitor sample, is shown in Figure 8. The spectrum was measured at the time of counting.

A correction for this error was determined by measuring a series of CCl_4 extractions at the time of the As^{76} count. Analysis of the spectrum indicated a possible error measurement between 0.7 and 0.9 Mev. A ratio of the count in this energy range versus the count in the As^{76} channel was determined for 15 separate random samples. The ratio of the two counting rates was $1.55 \pm 2\%$ and the small "standard" deviation indicates that a correction of this kind is valid. The spectrum of radioiodine isotopes is assumed to be constant during normal reactor operation. During an unusual operation, such as a rupture incident, the amounts of each iodine isotope might possibly change and the error correction would not be valid.

Since the ratio was determined at the exact time that a monitor count would have been made on the sample, the ratio can be used to determine the counting sequence for an interference correction. The ratio showed that there are 1.55 times as many error counts in the 0.56 Mev As^{76} channel

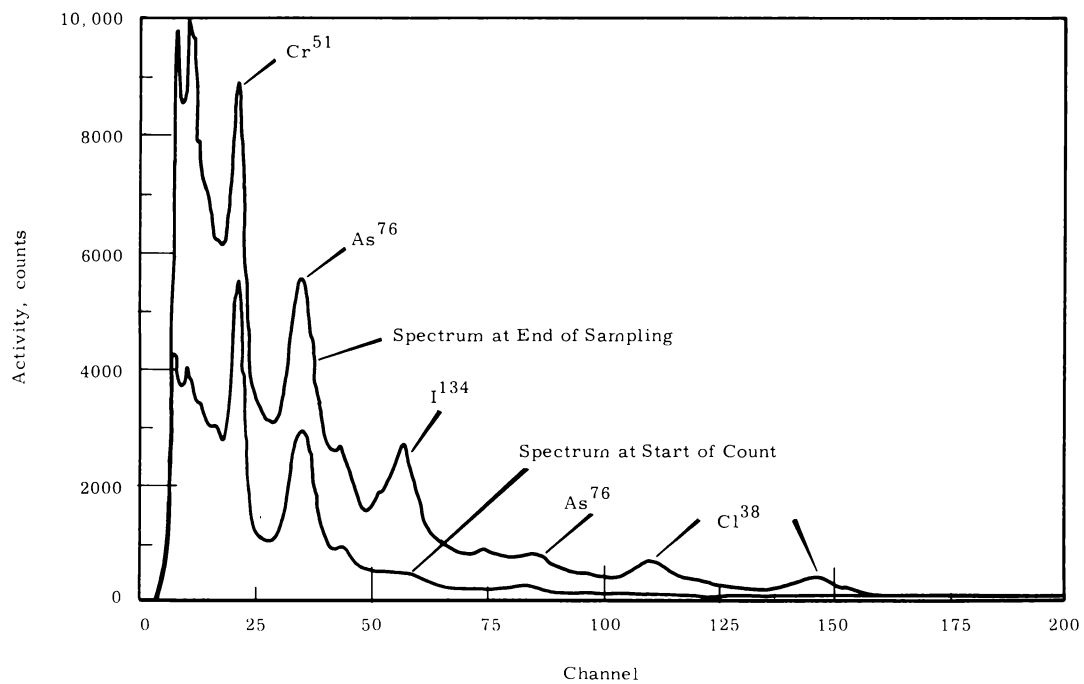


FIGURE 7
Gamma Spectrum of Column Effluent

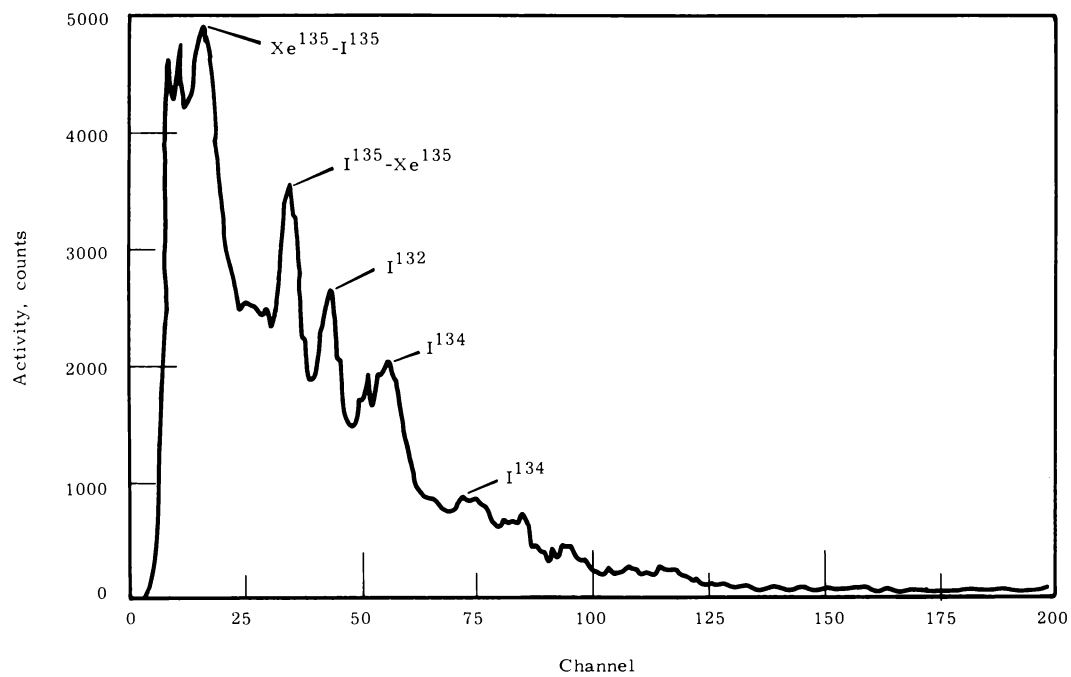


FIGURE 8
Spectrum of CCl₄ Extraction of Column Effluent

due to the iodine isotopes as there are in the 0.8 Mev correction channel. Therefore, a correction count is made by subtracting counts in the 0.8 Mev channel during a period 1.55 times as long as the forward count in the As⁷⁶ channel. Since this count also includes the background count in the correction channel, a background correction in the 0.8 Mev channel must be forward counted for the same counting time after the sample is drained from the cell.

Calibration

The monitor was calibrated by using standardized solutions of As⁷⁶ "spikes" prepared from irradiated arsenious acid in dilute HNO₃. The "spikes" were approximately the same concentration encountered in reactor effluent samples, and were calibrated by beta and gamma counting techniques. A conversion factor of 0.020 picocuries/ml/count/min was based upon 275 ml of sample and a 5 min counting time. The standard deviation for 10 calibrations was $\pm 5\%$.

Results from the monitor were checked by analytical methods and comparisons were made using analytical methods for As⁷⁶ determination and the monitor values were 10% high. Considerable difficulty was encountered in obtaining cross checks between the various analytical methods.

Reliability of Operation

Several periods of 2 wk operations were experienced with no attention to the monitor. During earlier operation, excessive temperatures in the cabinet caused gain shifts in the amplifier. Additional ventilation was used to overcome this problem. The scaler operation was reliable during most of the monitor operation; however, trouble was experienced in performing routine servicing requirements and improvements in the scaler circuit remedied this problem. Size of the ion column was increased to 500 ml of resin to increase the holdup time for Cl³⁸ decay and to reduce the frequency of column regeneration. Approximately 1 mo of operation can be obtained with one column. Some trouble was encountered during reactor shutdowns when cold water was fed to the column. This caused degassing from the sample within the resin and an occasional vapor lock

disrupted flow through the resin bed. A heating coil was installed upstream from the resin column to degas the water by preheating to approximately 70 C before passage through the column.

CONCLUSIONS

Concentrations of As^{76} in reactor effluent water can be measured by the As^{76} prototype monitor. With proper correction for isotope interference, the values should be within 10% of analytical results. Considerable variations in background can be tolerated; no significant change in As^{76} values resulted when the sample cell contamination was reduced from 550 to 125 counts/min. The minimum detection limit is 20 picocuries/cc. This value is two times the level of activity indicated by the monitor during periods of extended reactor shutdown.

The cost of the prototype instrument was approximately \$5000; the scaler, programmer, and printer were the major items of expense. The scaler and programmer were shop fabricated; the remaining electronic hardware were commercial items.

The maintenance and servicing requirements should average less than 2 hr/wk. The major servicing requirements are cation exchange column regeneration, pump tubing replacement, sample cell decontamination, and amplifier gain checks.

An undesirable feature of the prototype monitor method for As^{76} measurement is the requirement that the spectrum of interfering isotopes does not change during equilibrium operation. If, for example, the ratio of the various iodine isotopes would change due to an abnormal condition such as a rupture, the interference correction might not be correct. The correction factor at each reactor should be verified periodically by analytical methods.

ACKNOWLEDGEMENTS

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APPENDIX A

Programmer Sequence

The timing for sampling, counting, and data recording functions is regulated by circuits in a programmer unit. Five microswitches are actuated by a 1 revolution/hr timer. The timing of counting periods is regulated by two interval timers. The timing sequence is shown in Figure A-1.

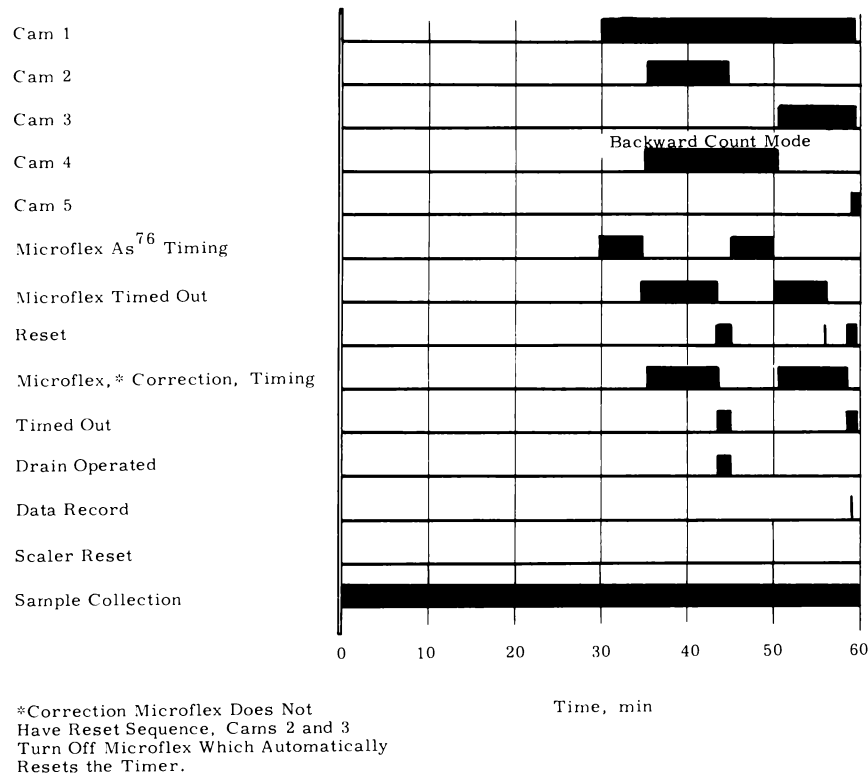


FIGURE A-1

Programmer Sequence

Cam Microswitch One energizes the As⁷⁶ timer which times a 5 min forward count in the As⁷⁶ channel. When the timer "times out," a relay switches the analyzer base line to the correction channel. Cam Microswitch Four energizes relays which cause the scaler to scale backward.

Cam Microswitch Two then energizes the correction timer which times an 8 min backward count in the correction channel. When the timer "times out," the As⁷⁶ timer resets and the sample drain solenoid valve is actuated. Reset of the As⁷⁶ timer deenergizes the base line relay and switches the analyzer to the As⁷⁶ channel. One minute later, Cam Microswitch Two deenergizes the correction timer. This causes the As⁷⁶ timer to start a 5 min backward count in the As⁷⁶ channel. When "time out" occurs, the base line is shifted to the correction channel. Cam Microswitch Four deenergizes the backward count relays.

Cam Microswitch Three then energizes the correction timer which times an 8 min forward count in the correction channel. When the timer "times out," the printer is energized and Cam Microswitch Five energizes a relay which initiates a print cycle. Cam Microswitches One and Four deenergize the timers. Cam Microswitch Five deenergizes the print relay and causes a stepping switch to step forward one position. This actuates solenoid sample valves which drain a new sample to the sample cell, and divert the column effluent to the empty reservoir. The stepping switch provides time coding information and the forward step increases the "hours" by one.

Scaler Driver Operation

The glow transfer tube requires two negative pulses of correct amplitude, duration, and timing for proper operation. The glow transfer or scaling action is caused by routing the pulses to Pins 11 and 12 of the glow transfer tube.

The two negative pulses are generated by four 2N398 transistors as shown in the scaler circuit diagram. TR-3, TR-4, TR-5, and TR-6 are normally conducting. When a positive input pulse is applied to the TR-3 base, it is cut off, causing a negative pulse at the emitter for the duration of the input pulse. TR-4 is connected to TR-3 as an emitter-follower, and the pulse is transmitted to Pin 12 of the glow transfer tube. The base of TR-5 is coupled to the emitter of TR-4 and when TR-4 begins to conduct, the trailing edge or positive going portion of the first pulse causes TR-5 to

cut off. The TR-5 emitter produces a delayed pulse which is applied to Pin 11 of the glow transfer tube through the emitter-follower, TR-6.

Pulse width or duration is controlled by the coupling capacitors and a 55 μ sec pulse gives satisfactory performance. A minimum overlap of 10 μ sec for the two pulses is recommended; however, the fast rise time of the second pulse made it possible to reduce the overlap to approximately 4 μ sec. The direction of counting is controlled by a relay which feeds the first pulse to Pin 12 for forward counting and to Pin 11 for backward counting. The relay also switches the second pulse to the opposite pin.

Carry pulses between decades are taken from the 0 or 9 cathode depending on the direction of counting. The appropriate selection is made by the same relay that controls the direction of counting for each decade. A relay contact in the circuit which furnishes the first pulse to the glow transfer tube prevents error counts from being registered during backward-forward switching sequences.

Scaler reset is accomplished at the start of the counting period by switching all cathode resistors from ground to +108 v except the 0 cathode. This action makes all cathodes except the 0 cathode positive and the anode to cathode glow transfers to the 0 cathode.

APPENDIX B

PROGRAMMER CHASSIS CIRCUIT DIAGRAM

QTY.	PT. NO.	DESCRIPTION
1	1	PROGRAMMER - SEE SHEET 2
1	2	PRINTER-SCALER SEE SHEET 3
1	3	HIGH VOLTAGE SUPPLY - ATOMIC INST. CO. MODEL 312
1	4	COUNT RATE METER - SEE SHEET 3
1	5	AMPLIFIER-ANALYZER - RADIATION INSTRUMENT DEVELOPMENT LABORATORY - MODEL DD-2
1	6	BLOWER
1	7	RELAY CABINET RACK - BUD CR-2282, 82 1/2" H x 22" W x 22" D
1	8	COLUMN HEAD TUBE
4	9	SAMPLE RESERVOIR
1	10	RESIN COLUMN
4	11	SOLENOID VALVE - AUTOMATIC SWITCH CO. NO. B2626
4	12	SAME AS PART 11
1	13	SAME AS PART 11
1	14	DRAIN FUNNEL
1	15	JUNCTION BOX - 5" W x 10" L x 2" D
1	16	LEAD SHIELD - 12 8"x4"x3" 1/2" 6 8"x4"x2" LEAD BRICKS.
1	17	SAMPLE CELL
1	18	SODIUM IODIDE CRYSTAL - HARSHAW 3" x 3" D
1	19	PHOTOTUBE - DUMONT 6363 F PREAMPLIFIER - SEE SHEET 3
1	20	PUMP - SIGAMOTOR MODEL T-8

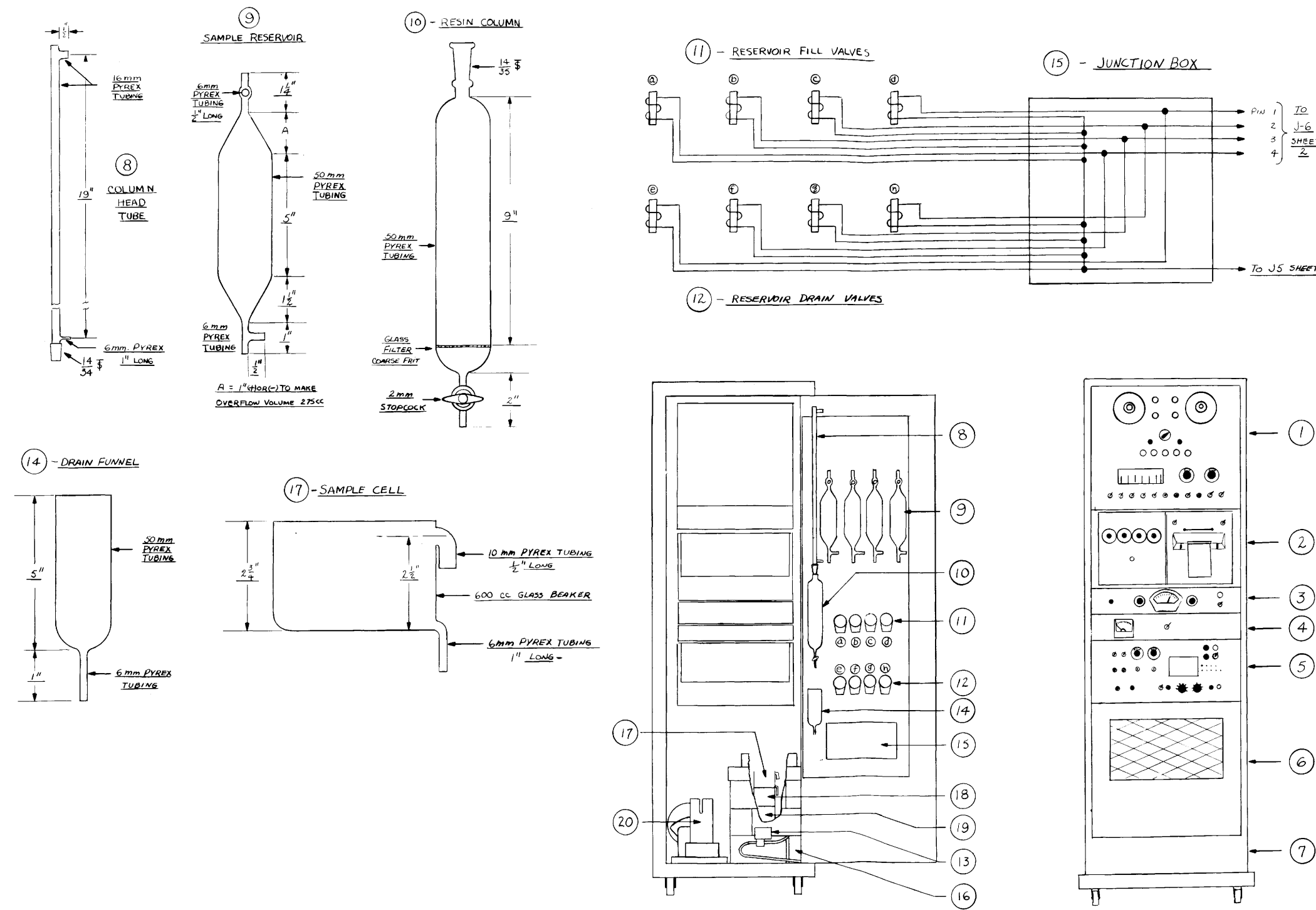
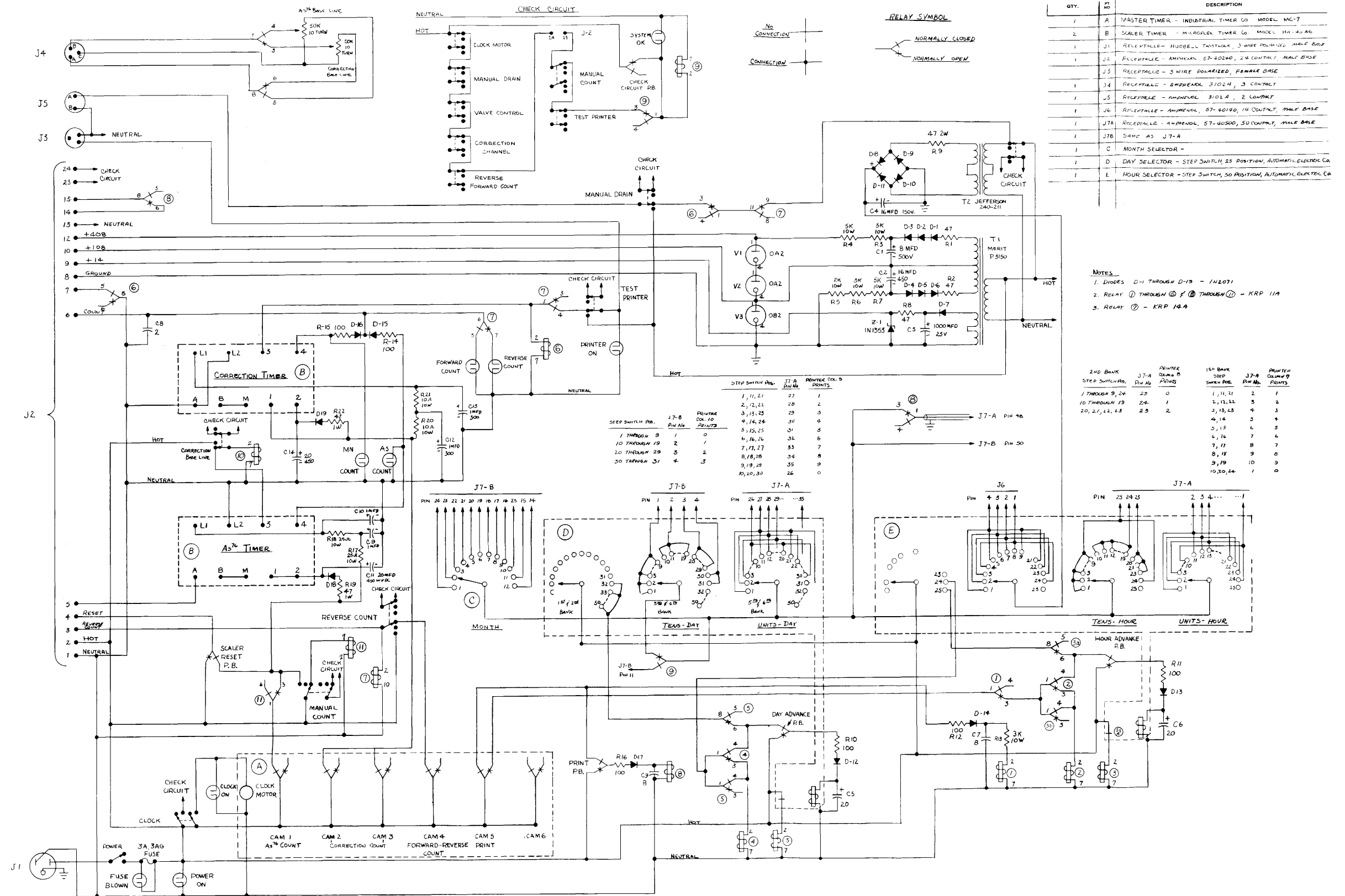


FIGURE B-1

As⁷⁶ Monitor Assembly

APPENDIX C

SCALER CIRCUIT DIAGRAM



APPENDIX D

SAMPLING SYSTEM COMPONENTS

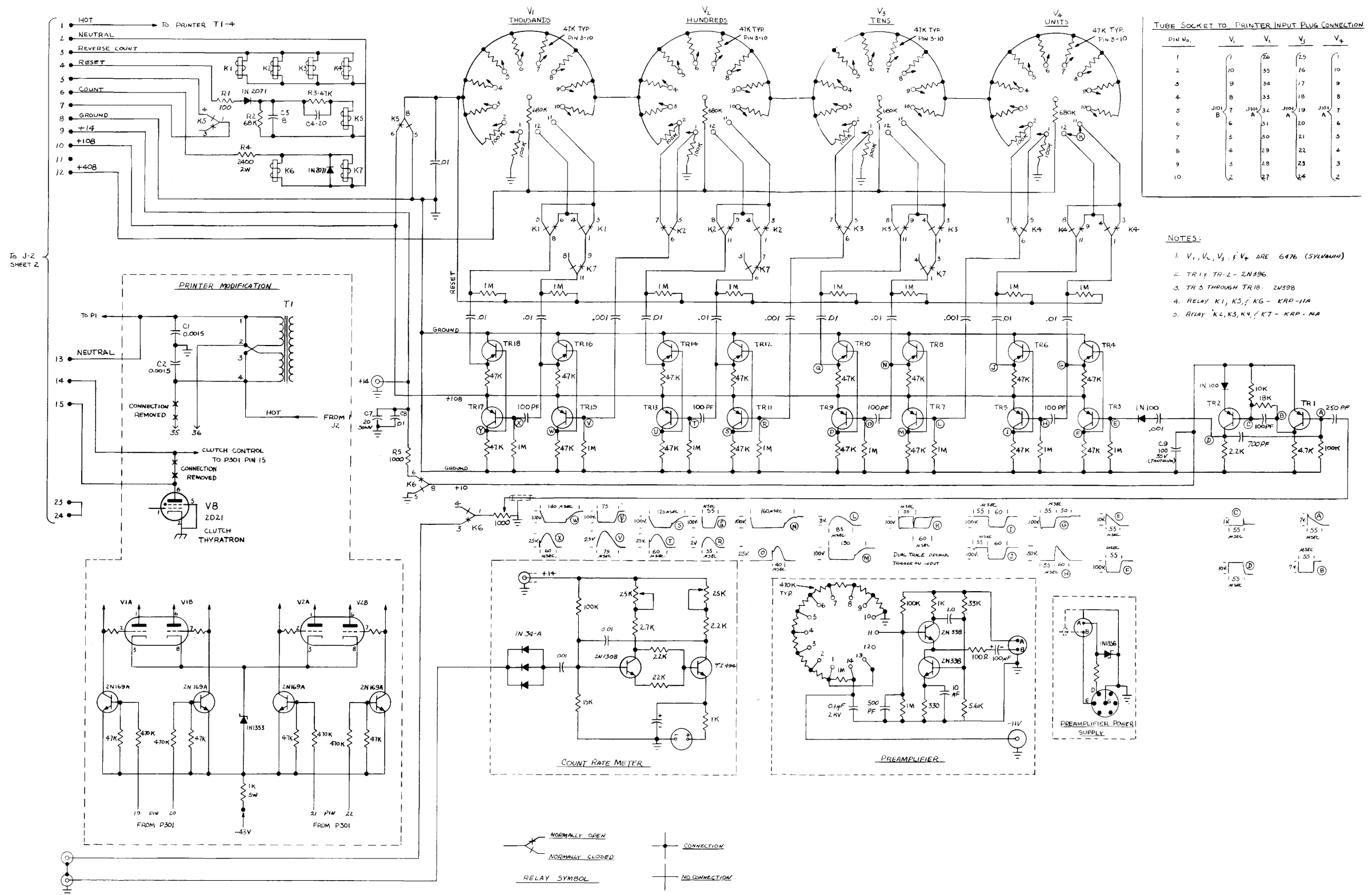


FIGURE D-1

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