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ATOMIC ENERGY COMMISSION

ELECTRONICS FOR PROCESSING OF DATA FROM A DOUBLE COLLECTOR ISOTOPIC RATIO MASS SPECTROMETER

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A B S T R A C T

The output data available from the mass spectrometer type MS-660 developed in the mass spectrometry section of Technical Physics Division for the determination of H/D ratios in liquid/gas sample consists of uncompensated mass 3 and mass 2 signals. After the mass 3 signal has been compensated for H_3^+ formation, the on-line ratio of compensated mass 3 to mass 2 is calculated, displayed, and then printed on a printer for record.

The present work explains the electronic compensation circuit, the discrete voltage-to-frequency (V/F) converter circuit, the ratio calculating system using V/F converters, and a digital interface system for Hindustan Teleprinter to print out the ratios. Results obtained on mass spectrometer MS-660 are presented.

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INTRODUCTION

The mass spectrometer MS-660 developed in the Technical Physics Division is a double collector type machine with a radius of 6 cms. and 60° magnetic deflection geometry. The main function of the machine is to provide an important facility of direct ratio determination in water samples in a wide range of applications like heavy water analysis, hydrology work etc. When liquid or gas samples are injected into the sample handling system of the mass spectrometer, the separated isotopic current signals constituting mass 3 and mass 2 are collected simultaneously at the two collectors and converted into voltage signals by highly stable d.c. amplifiers. These signals are ready for further processing.

(i) Electronic H_3^+ compensation :

The output of the mass spectrometer should read out the ratio of mass 3 viz. HD^+ to mass 2 viz. H_2^+ signals. However, the mass 3 signal available at the amplifier output has two mass 3 components : HD^+ and H_3^+. H_3^+ is formed according to the following ion-molecular reaction:

$$H_2^+ + H_2 \longrightarrow H_3^+ + H$$

Since $H_2^+ \propto P$, where P is the pressure :

$$H_3^+ \propto P^2$$

or $$H_3^+ \propto (\text{ Ion current mass 2})^2$$

or $$H_3^+ = K \ (\text{Ion current mass 2})^2$$

If correction for H_3^+ formation is not provided, then instead of (HD^+/H_2^+) ratio, the machine will read $(HD^+ + H_3^+)/H_2$. In order to get the ratio (HD^+/H_2^+) only, the post ratio correction has to be applied which is rather very inconvenient and time consuming. Therefore, in our machine this correction has been applied before the ratios are determined. Fig.(1) gives the circuit for the electronic compensation. The multiplier MC 1494 squares the mass 2 signal. A small signal proportional to the squared mass 2 signal is then substracted from the mass 3 signal. The output of the subtractor mono op-7 amplifier is the compensated mass 3 signal $(M_3 - KM_2^2)$. The factor K depends upon the formation of H_3^+ with respect of mass 2 values. Its typical value is 3 ppm/volt of mass 2 for natural water concentration. However, the value varies with the sample concentration. A front panel potentiometric control sets the value of K.

(ii) Ratio measuring System:

There are several ways of determining ratio of two analog signals. For example, using analog multiplier/divide modules, log/antilog modules, digital multimeters with ratiometric option, voltage to frequency (V/F) convertors, binary rate dividers etc. In our application ratio measuring system using V/F converters has been developed and incorporated. Commercially available V/F converters have very high linearity (0.002%), very low resolution, wide dynamic range, monotonicity and excellent noise rejection.

A V/F converter using discrete compenents was developed. This circuit is based on the charge-balancing method (1). The circuit is given in Fig. (2). The specifications obtained from this circuit are :

Input Voltage range	:	0 to + 8V
Output frequency	:	5 KHZ/volt.
Non-linearity (from 200 mv to 8V)	:	0.1%

The reason for these not so good specifications are the incompatability of discrete components, low gain of the integrator, absence of correcting feedback loop, voltage and current offsets etc. For highly precise ratio measuring system these voltage to frequency converters are not suitable. However, for mass 2 measurement this V/F converter was converted into a digital panel meter with 1 sec. sampling rate. This has been incorporated in the mass spectrometer.

The commercial available V/F converters type 4715 made by Teledyne Phil brick of USA were used. These converters have the following typical specifications.

Input voltage range	:	0 to ± 13V
Frequency range	:	0.01 HZ to 13 KHZ
Non-linearity	:	0.002% .

Using the symmetrical V/F converters, a ratio measuring system has been developed. The system is described in fig. (3). The principle is as follows :

Let f_1 (corresponding to mass 3 signal) = x pulses/sec.

f_2 (corresponding to mass 3 signal) = y pulses/sec.

$$f_1 < f_2 \quad \text{or} \quad f_1 > f_2$$

For time t secs. :

$$f_1 = x.t \text{ pulses}$$

$$f_2 = y.t \text{ pulses}$$

When y.t = M, the reference value,

$$\text{Ratio} = \frac{x.t}{M} = \frac{x.t}{y.t} = f_1/f_2 = \frac{\text{Mass 3 signal}}{\text{Mass 2 signal}}$$

The ratio f_1/f_2 equals the ratio of input voltages to the V/F converters. The ratio accuracy depends primarily upon the accuracy of V/F converters. Fig. (4) gives the complete circuit details. The ratio is displayed by 5 LED displays. Fig. (5) gives the respective wave-forms of outputs A, B, C, D, E and $\overline{C}$ in the frequency division.

The two most important factors one has to keep in mind while using these highly linear V/F converters are the grounding problems and the calibration. The calibration becomes doubly important for accuracy because of the need for the use of two similar converters. In this work, the calibration was done using highly stable (30 ppm) d.c. source, 5½ digit Fluke DVM and 10 MHZ frequency counter. Enough care was taken to make all the ground points to be near zero potential. The results obtained using the ratio measuring system alongwith the H_2^+ compensation are tabulated in Tab. (1).

The system can also be used in ratio and integrate mode with a variable integrating time. In the ratio mode only, the system will be as described above. In the ratio and integrate mode, the overall system will be same with the only change in the reference input value. This value is constant in the case where ratio is only calculated. However, when signals are to be integrated besides calculating the ratios, the reference input value is made variable. Digital integration for any length of time can be performed and ratios of integrated signals displayed. This also has been incorporated in the mass spectrometer with a front panel control which puts the system in ratio mode only or integrate and ratio mode.

(iii) Digital Ingerface for Hindustan Teleprinter:

In order to have a permanent record of the processed data e.g. the ratios, from the mass spectrometer, the Hindustan Teleprinter was used. However, the data available from the mass spectrometer is in the parallel BCD form whereas the teleprinter accepts the data only in the serial Elliot 803 code. The interface system described here accepts the parallel BCD code, converts it to the Elliot 803 code and feeds it to the Teleprinter where the date is printed. The data can be printed upto 6 digits by this system.

System Description:

Read only memories are being used extensively for the code conversion in the digital systems. This application idea has been made use of in the development of interface system. Fig. (6) describes the system diagram.

The Elliot 803 code which is accepted by the teleprinter is wired permanently in the ROM, Fig. (7). Since our application requires only the printing of digits and a decimal, the codes for the digits 0 to 9 and codes for teleprinter operation like carriage return, line feed and figure shifts are put in the ROM. Then this ROM is addressed by the address generator. The address generator Fig. (8), generates the address from the data input and precision clock pulses. The parallel output from the ROM is then converted in the serial form using the parallel-to-serial converter, Fig. (7), which is addressed by the proper address lines at the proper instant of time. The serial output from the convertor is effectively the Elliot 803 code for either a digit or a particular operation of the teleprinter. It is fed into the teleprinter current loop through a switching transistor. Fig. (9) shows the way the data is fed.

Though the interface has been developed to print out the processed data from the mass spectrometer, it can in principle be used to print any data available in BCD form. In the present application only digits have been printed. However, the same technique can be extended to print the numerals also.

CONCLUSIONS

The electronics system developed to process data from mass spectrometer has been described. The system provides the compensation for H_3^+ formation and calculates on - line the ratio of compensated mass 3 and mass 2. Facility has also been provided to print out the data on Hindustan Teleprinter. If required the signals can also be digitally integrated before the ratios are calculated.

With the incorporation of the processing electronics in the mass spectrometer, the time of analysis has been considerably reduced. It has also reduced the strain on the operator operating the mass spectrometer. From the Table (2) we notice that the integrated precision values is not very much different from unintegrated valies. However, the operator errors are avoided when the processing electronics is used in ratio and integrate mode. But the time of analysis is more in the later case.

Though the system has been developed for mass spectrometer, it can be made use of in any system where two d.c. signals within $\pm$ 10 volts are available simultaneously.

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<u>REFERENCES</u>:

1. Designer's Guide to V/F Converters, 1975 by DATEL SYSTEMS, INC. of USA.

TABLE (1)

Results obtained on mass spectrometer MS-660 using on-line ratio measuring system with H_3^+ compensation (multiple injection).

(i) **Analysis with distilled water sample (ratio mode only)**

Mass 2 (Volts)	Ratio
7.34	0161.7
6.64	0161.8
6.00	0161.8
5.40	0161.7
4.85	0161.8

(ii) **Analysis with (approximate) 330 ppm water sample (ratio mode only)**

Mass 2 (Volts)	Ratios
7.4	0332.9
6.54	0333.0
5.82	0333.0
5.20	0333.0
4.68	0333.0

(iii) **Analysis with water sample (integrate + ratio mode)**

Mass 2 (volts)	Ratio
7.35	157.58
6.53	157.55
5.82	157.57

TABLE (2)

Results of the precision obtained on MS-660 using single injection of the sample.

Sample : Natural Water.

Injection	Rel (%) (ratio mode)	Rel (%) (Ratio + integrate mode
First	0.07	0.04
Second	0.03	0.03
Third	0.03	0.02
Fourth	0.03	0.015
Fifth	0.032	0.02
Sixth	0.03	0.015
Seventh	0.03	0.03
Eighth	0.01	0.028
Nineth	0.05	0.033
Tenth	0.05	0.024
Average rel (%)	0.028	0.0226

* Integration time : About 16 seconds.

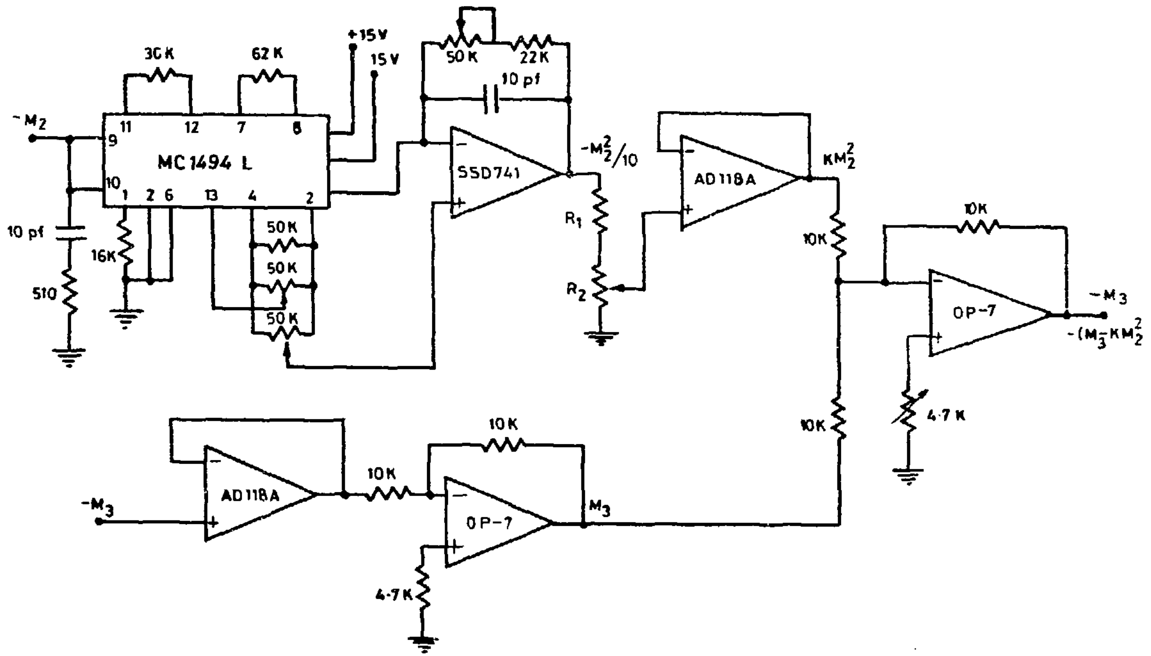

FIG. 1 : COMPENSATION CIRCUIT

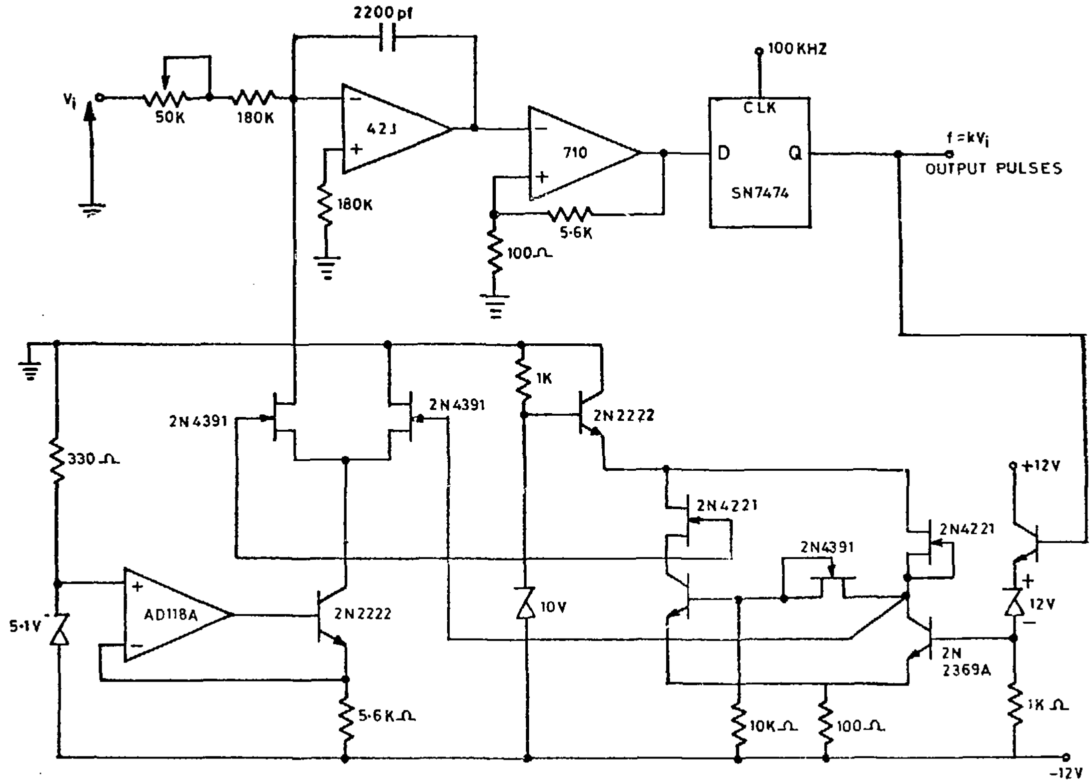

FIG. 2 : VOLTAGE TO FREQUENCY CONVERTER

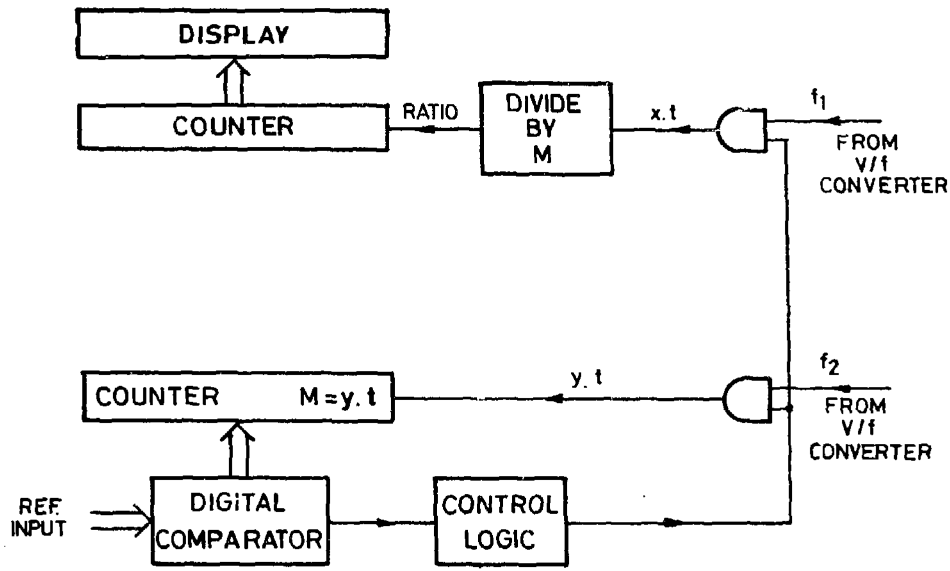

FIG.3: FREQUENCY DIVISION SYSTEM

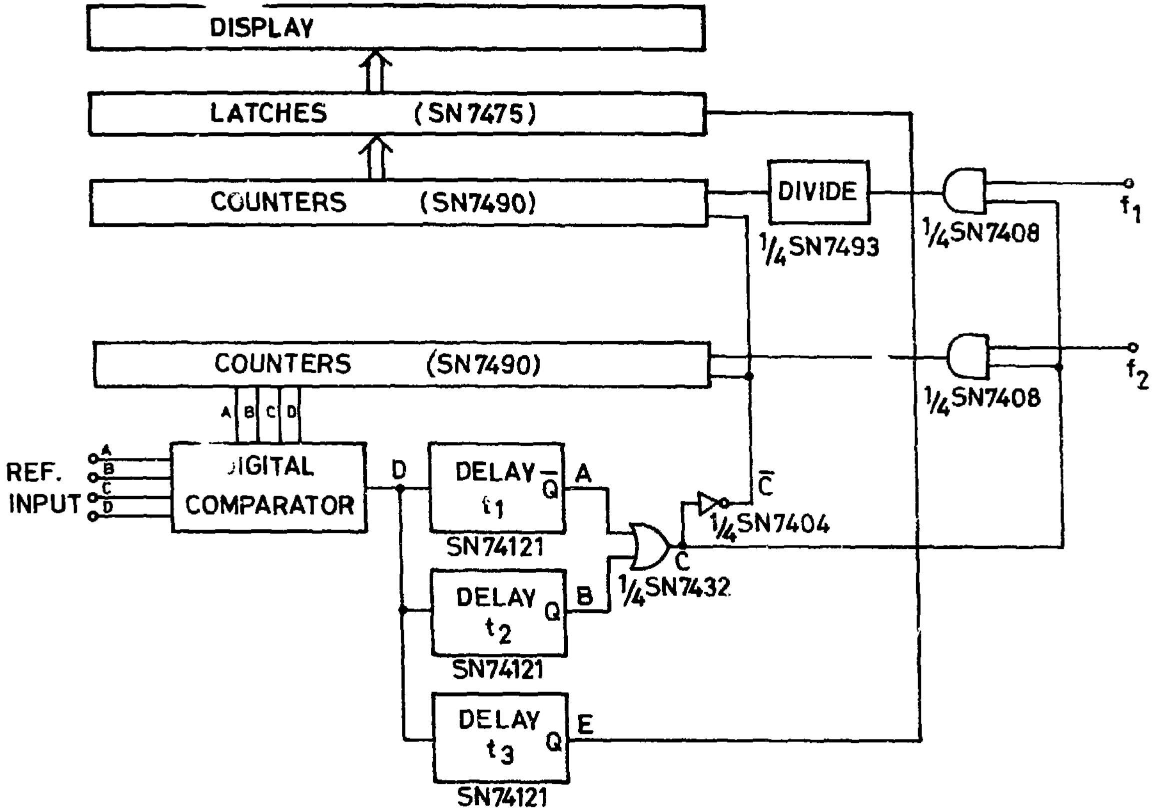

FIG. 4 FREQUENCY DIVISION CIRCUITS

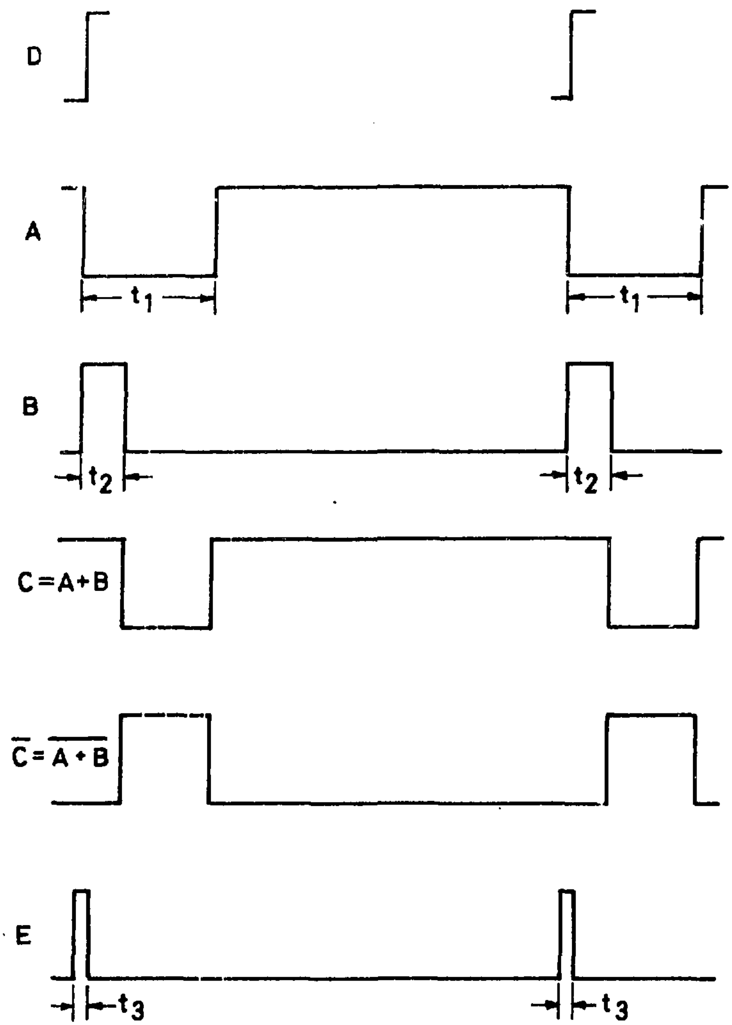

FIG.5 : WAVEFORMS OF OUTPUTS A, B, C, D, E AND $\overline{C}$ IN FREQUENCY DIVISION CIRCUIT

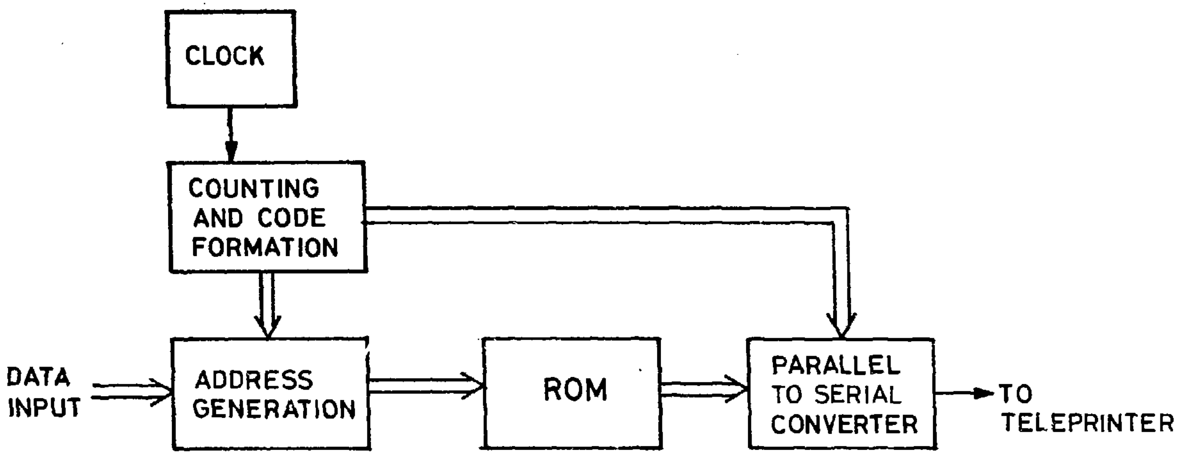

FIG. 6 : INTERFACE SYSTEM DIAGRAM

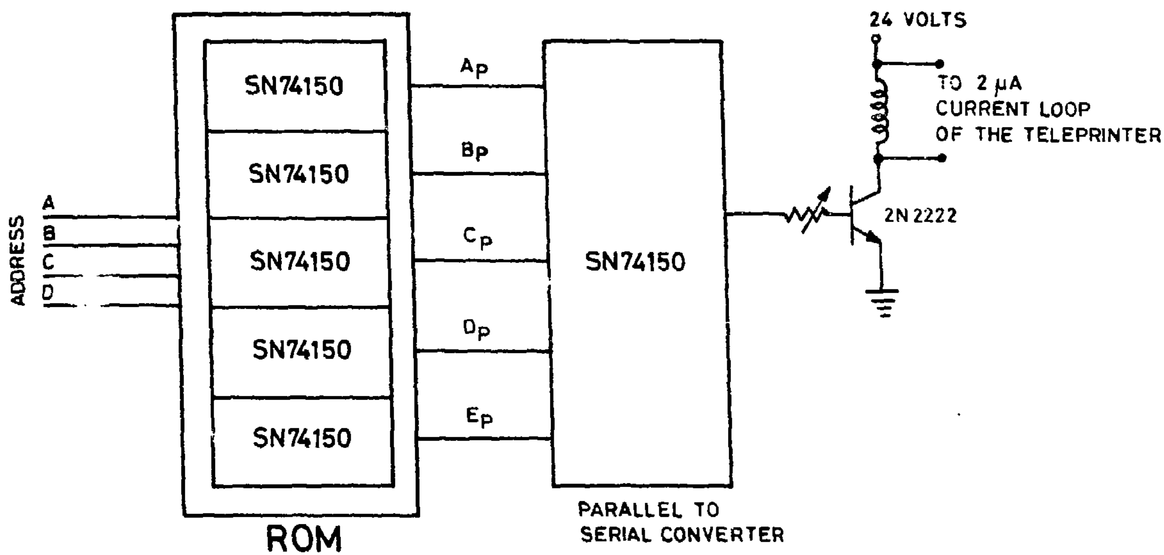

FIG. 7: MULTIPLEXERS CONNECTED AS ROM AND PARALLEL TO SERIAL DATA CONVERSION

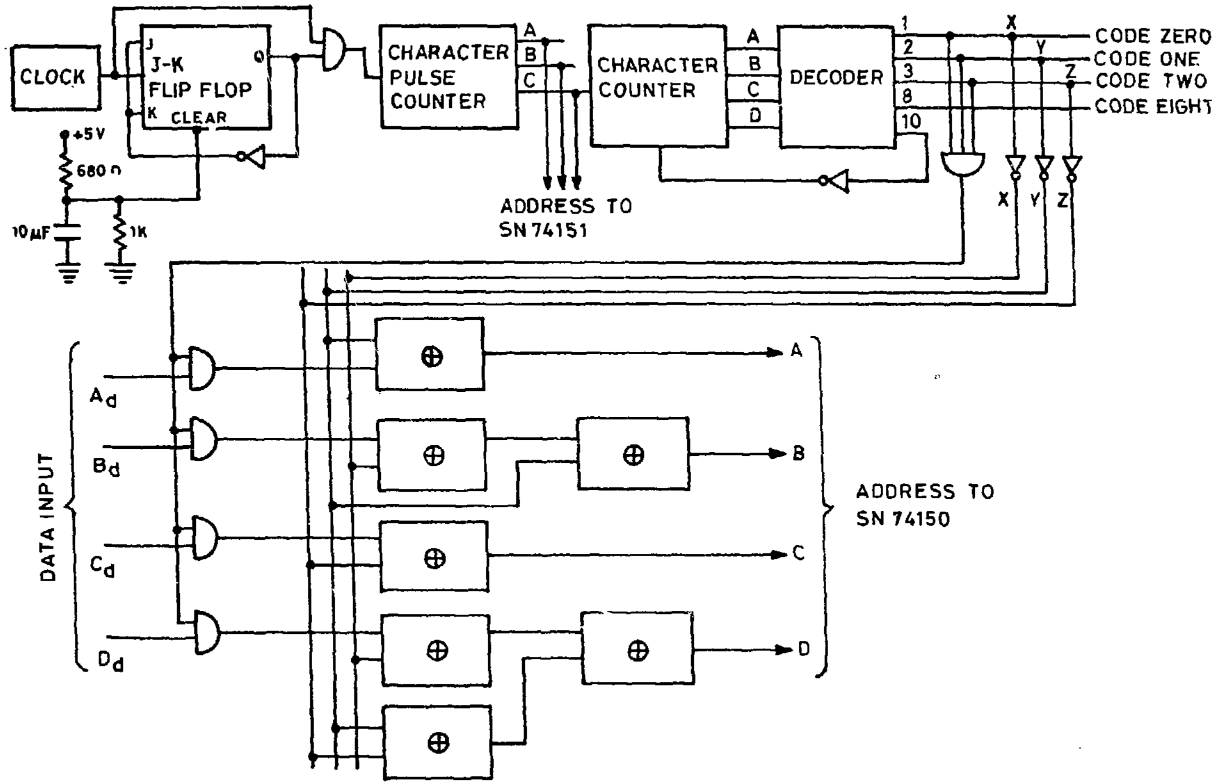

FIG. 8 : ADDRESS GENERATION

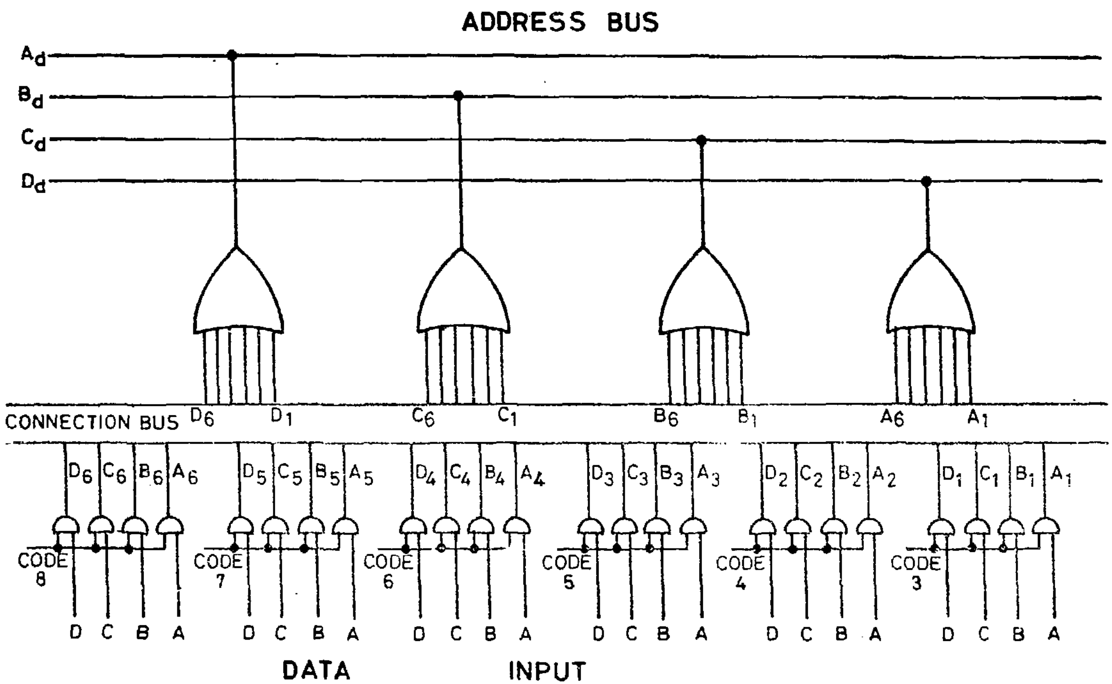

FIG. 9 DATA INPUT