Thermochimica Acta, 51 (1981) 85—95
Elsevier Scientific Publishing Company, Amsterdam — Printed in The Netherlands

ON AN IC ELECTRONIC BALANCE CONTROL SYSTEM AND ITS IMPACT ON THE CALIBRATION

L. BINARD
Electronics and Instrumentation Service, S.C.K./C.E.N., B-2400 MOL (Belgium)
A. VAN DEN BOSCH, J. VANSUMMEREN
Materials Science Department, S.C.K./C.E.N., B-2400 MOL (Belgium)

ABSTRACT

The contribution deals with an "1974 integrated circuit" version of an electronic damping and nulling system, adapted to a torsion balance which now is more than twenty years old. The discussion of calibration results reveal that masses of about 0.5 mg, in certain cases, can be intercompared with a relative precision of 3×10^{-5}, indicating that the balance sensibility is better than 2×10^{-8} g. As the deflection sensitivity of the balance is 1.3×10^{-4} deg/μg the position sensor on the balance can detect, over a few minutes, average displacements of about 4×10^{-7} cm (i.e. ~ 40 Å).

INTRODUCTION

An automatic recording torsion balance has been presented at the fifth conference on Vacuum Microbalance Techniques by Van den Bosch (ref. 1), who reported briefly on the part of his thesis (ref. 2), concerning the apparatus built for static magnetic susceptibility measurements. The need for reliable susceptibility values together with the nature of the constructed balance, required the development of an electronic damping and nulling system. A vacuum-tube system was ready in November 1959; its improved version of July 1960 practically was not changed anymore. Joule heating, however, slowly degraded some components. A more stable version was searched for in the semiconductor technology. The final "Integrated Circuit" system, dealt with in this contribution, replaced the vacuum-tube system in July 1974 and improved the force measurements.

THE "INTEGRATED CIRCUIT" CONTROL SYSTEM

The block diagram, showing the scheme for automation of the microbalance with the use of integrated circuit components, is given in Fig. 1. The linear differential

0040-6031/81/0000—0000/$02.75

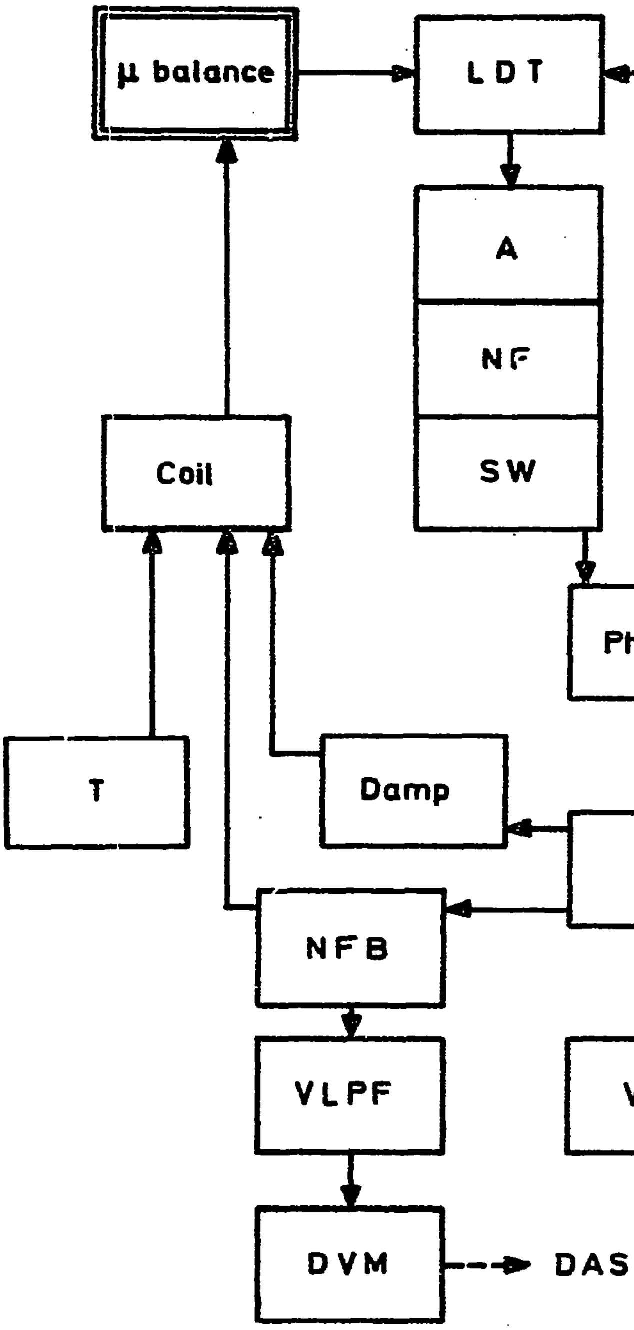


Fig. 1

Microbalance control system block diagram.

transformer (LDT), sensing the microbalance beam position, is the same which was used before in the old vacuum-tube balance control version. The LDT 8 kHz output signal is fed into an a.c. differential amplifier (A). High frequency noise and parasitic signals deliberately are filtered out (NF). The main signal is further amplified and shapened into a square wave (SW). A reference square wave (RSW) is formed from a phase adapted signal which is derived from the oscillator-(Osc) output that energizes the position sensor. Both square waves are fed into a logic exclusive-OR gate for phase detection (Ph.Det). The detection output is sent through a ripple eliminating filtre (F), yielding a signal that is used for further amplification but also for a vibration level indicator (VLI). The importance of this VLI is explained under "Results and Discussion". The main signal generates a voltage resulting in the negative feed-back current (NFB) which keeps the balance beam continuously at the null position. The same voltage, filtered out by a very low pass filter (VLPF), is measured by the digital voltmeter (DVM) of the data acquisition system (DAS) (ref. 3). The capacitor of the damping circuit (Damp.) is fed by the main signal through a frequency-adapter. A three position switch, 10 mg/1 mg/0.1 mg, is inserted in the NFB-circuit, in order to facilitate the use of the apparatus.

Compared to the 1960 vacuum-tube control system the 1974 integrated circuit version damps the balance more critically and nulls it faster. The high reliability of the latter version allowed the balance to be controlled practically continuously for more than six years now.

RESULTS AND DISCUSSION

The calibration of the force meter is based on the comparison of the negative feed-back balance control output voltage difference to the mass of a reference weight in the earth's gravitational field. A mechanism for putting one of two weights on the balance has been built in at the early stage of the development of the data logging system, with the appearance of a more precise voltmeter in our susceptibility apparatus in October 1968. Both reference weights are rings of molybdenum wire. The diameter of the wire for the heavier mass, m_1, is 50 µm; that of the other mass, m_2, is 40 µm. The mass of the smaller one is about 0.5 mg.

Calibration results, for the period enclosing the replacement of the vacuum-tube (VT) balance control system by the integrated-circuit (IC) version, are given in Table 1. The data in column 3 and 4 refer to averages of the balance system output voltage differences V_2 and V_1 which are related respectively to the masses m_2 and m_1. In column 5 are given the ratios $R = \bar{V}_1/\bar{V}_2$. The data have been taken at room temperature T = 295 (± 2) K, with the digital voltmeter Dynamco 2022.

The average of the ratios, calculated from data taken in the last three months of 1973, is $\bar{R}$ = 1.7112. The σ value (ref. 4) on this fig. is 40 x 10^{-4} yielding a variation coefficient $\zeta = (\sigma/\bar{V}) \leq 0.24$ %. In the first half of the year 1974 the values of the various parameters were as follows: $\bar{R}$ = 1.7068, $\sigma \leq 27 \times 10^{-4}$ and

TABLE 1

System	Date	$\bar{V}_2$	$\bar{V}_1$	R	
VT	22.10.73	−2.7233	−4.6702	1.7149	
	5.11.73	−2.7277	−4.6741	1.7135	
	12.11.73	−2.7267	−4.6687	1.7122	
	20.11.73	−2.7253	−4.6460	1.7047	
	18.12.73	−2.7216	−4.6556	1.7106	
	average	−2.7249	−4.6629	1.7112	$\sigma \leq 0.0040$, $\zeta \leq 0.24$ %
VT	6.02.74	−2.7222	−4.6443	1.7060	
	28.02.74	−2.7226	−4.6437	1.7056	
	9.04.74	−2.7241	−4.6511	1.7073	
	11.04.74	−2.7244	−4.6467	1.7055	
	29.04.74	−2.7237	−4.6631	1.7120	
	14.05.74	−2.7215	−4.6400	1.7049	
	average	−2.7230	−4.6481	1.7068	$\sigma \leq 0.0027$, $\zeta \leq 0.16$ %
IC	8.07.74	−2.6045	−4.4606	1.71265	
	23.07.74	−2.6045	−4.4606	1.71265	
	4.09.74	−2.6097	−4.4693	1.71257	
	9.09.74	−2.6104	−4.4707	1.71264	
	23.10.74	−2.6093	−4.4681	1.71237	
	3.12.74	−2.6110	−4.4710	1.71237	
	average	−2.6082	−4.4667	1.71254	$\sigma \leq 0.00014$, $\zeta \leq 0.008$ %

$\zeta \leq 0.16$ %. The relative difference between both $\bar{R}$ values is less than 0.26 % which is typical for the precision that was reached with the vacuum-tube balance control version. The data taken with the integrated circuit version of the balance control in the second half of the year 1974 were: $\bar{R} = 1.71254$, $\sigma \leq 1.4 \times 10^{-4}$ and $\zeta \leq 0.008$ %. The latter $\bar{R}$ value deviates from the previous one by only 0.34 % indicating that the proportionality of the output voltages to the masses holds rather well for both control systems. Nevertheless, although care has been taken not to change any other parameter, the replacement of the VT version by the IC one resulted in a difference between the $\bar{V}$ values of the first- and those of the second-half of the year 1974 by as much as 4.4 %. The effect is not due to a misfit of the NFB current-determining resistor's values as those were selected to be the same to within 0.1 %. At the introduction of the IC component system a decrease of the signal is observed. The explanation for the observed difference is sought in the hysteresis which occurs upon twisting the torsion ribbons (ref. 1) together with the slow maximum nulling speed, 0.5 mg per minute, of the VT balance control system. The idea is that when a reference ring is put on the balance the force is abruptly induced on the beam and the balance is out of control for a substantial fraction of a minute. When this interpretation is correct, then the 4.4 % difference should not be observed at the susceptibility measurements because, since July 1963 the power supply of the electromagnet is driven by a motor in such a way that the magnetic force on the sample is built up progressively to its maximum in about one minute. In agreement with the expectation, the relative difference $[\Delta_r = (V_{(VT)}-V_{(IC)})/V_{(IC)}]_{I_M}$, for a certain current intensity I_M at the energization of the electromagnet, is much smaller than 4.4 % at the susceptibility measurements. The relation holds irrespective of the sample being diamagnetic or paramagnetic as deduced from Tables 2 and 3 respectively. The agreement strengthens the interpretation that the faster the nulling circuit is, the more closely the torsion ribbons are kept to their null position so that, for a certain force, the output voltage difference will be less dependent on the induction rate of this force. It therefore is believed that the 1974 IC version of the balance control system allows the comparison of the magnetic induced forces to the reference forces to much better than 4.4 %.

TABLE 2

Sample holder, AuCu alloy T = 294.0 (± 0.4) K

System	I_M(A)	$\bar{V}_S$(V)	$\sigma.10^4$(V)≤	$\zeta.10^4$≤	$\Delta_r.10^4$
VT	1.8	+1.4497	7	5	
	1.4	1.3591	2	2	
	1.0	0.9529	13	14	
	0.8	0.6267	6	10	
	0.6	0.3474	5	15	
	0.4	0.1471	3	16	
IC	1.8	+1.4483	6	4	+10
	1.4	1.3566	5	4	+19
	1.0	0.9532	8	8	− 4
	0.8	0.6236	13	20	+50
	0.6	0.3457	3	7	+50
	0.4	0.1448	4	24	+159

TABLE 3

Sample 643, Al (0.57 at % Mn) converted to T = 297.0 K

System	I_M(A)	$\bar{V}_t$(V)	$\sigma.10^4$(V)≤	$\zeta.10^4$≤	$\Delta_r.10^4$
VT	1.8	−2.2055	5	3	
	1.4	−2.0824	10	5	
	1.0	−1.5026	−	−	
	0.8	−1.0169	9	9	
	0.6	−0.5962	16	27	
	0.4	−0.2774	4	13	
IC	1.8	−2.2069	5	3	− 7
	1.4	−2.0832	8	4	− 4
	1.0	−1.5003	9	6	+16
	0.8	−1.0169	7	6	−
	0.6	−0.5957	6	10	+ 9
	0.4	−0.2777	2	7	−11

A remarkable performance of the integrated circuit version of the balance control is the small value of the variation coefficient on R : $\zeta \leq 1 \times 10^{-4}$. The relative precision on the measurements, considered over more than four years, is about twenty times better than that obtained with the vacuum-tube system. A contribution to this improvement of the reproducibility is due to the introduction of the vibration level indicator. Indeed, the device allows for controlling the vibration level in the laboratory. The level is kept as low as possible during the calibration of the balance.

In the period 1974-1978 the IC control system yielded in fact a relative precision which is practically that claimed for the digital voltmeter in use. The coincidence raised the question whether or not the reproducibility of the ratio measurements, carried out with the aid of the IC version, is limited by the precision of the voltmeter. The data given in Table 4 seem to indicate that indeed this is the case as the introduction, in April 1978, of the Solartron 7075 digital voltmeter, having a precision in the 0.1 sec integration mode of about 3×10^{-5}, yields this figure for the variation coefficient on the reference weight's signal ratios.

TABLE 4

DVM	Date	$\bar{\bar{V}}_2$(V)	$\bar{\bar{V}}_1$(V)	$\bar{R}$	
Dynamco 2022	1974	-2.6082	-4.4667	1.71254	
	1975	-2.6122	-4.4728	1.71227	
	1976	-2.6135	-4.4753	1.71236	$\bar{\bar{R}}$ = 1.71250
	1977	-2.6145	-4.4777	1.71264	σ < 0.00019
	1978	-2.6149	-4.4786	1.71272	$\zeta \leq$ 0.012 %
Solartron 7075	1979	-2.6148	-4.4784	1.71271	$\bar{\bar{R}}$ = 1.71276
	1980	-2.6151	-4.4791	1.71278	σ < 0.00005
	1981	-2.6142	-4.4776	1.71280	$\zeta \leq$ 0.003 %

CONCLUDING REMARKS

This paper deals with an electronic balance control system that has been built in 1974 with integrated circuit components for an old torsion balance. The adjustment of the damping and nulling circuit improved the balance performances in a remarkable way. The ratio of two output voltage differences, related to two reference weights, was before reproducible to within 0.6 % over a period of one year. The introduction of the IC system increased the reproducibility by a factor about twenty. The extreme values of the ratios, considered over a period of six years, did not differ by more than 0.031 %. The use of a more precise voltmeter seems to reduce the uncertainty even more. The extreme values have been kept as close as 0.006 % over a period of two years.

The discontinuity in the output voltage difference, related to a single reference weight, versus time at the introduction of the IC version indicates that the faster nulling in the latter version keeps the difference in output signal small when the same force is induced on the balance at a different rate.

The remarkable gain in precision with the use of the 1974 electronics resulted in a sensibility of the balance system of 3×10^{-5} times the smaller reference mass, $m_2 \simeq 0.5$ mg, i.e. a sensibility better than 2×10^{-8} g.

In Schwoebel's survey (ref. 5) on the characteristics of beam microbalances, utilizing horizontal suspension fibers, is the deflection sensitivity of the balance considered here, 1.3×10^{-4} deg/µg, by far the smallest. The sensitivity is equivalent with a displacement of the core of the linear differential transformer of 2.2×10^{-5} cm/µg. The sensibility of $\leq 2 \times 10^{-2}$ µg consequently relates to an average core displacement of $\leq 4 \times 10^{-7}$ cm (i.e. ≤ 40 Å).

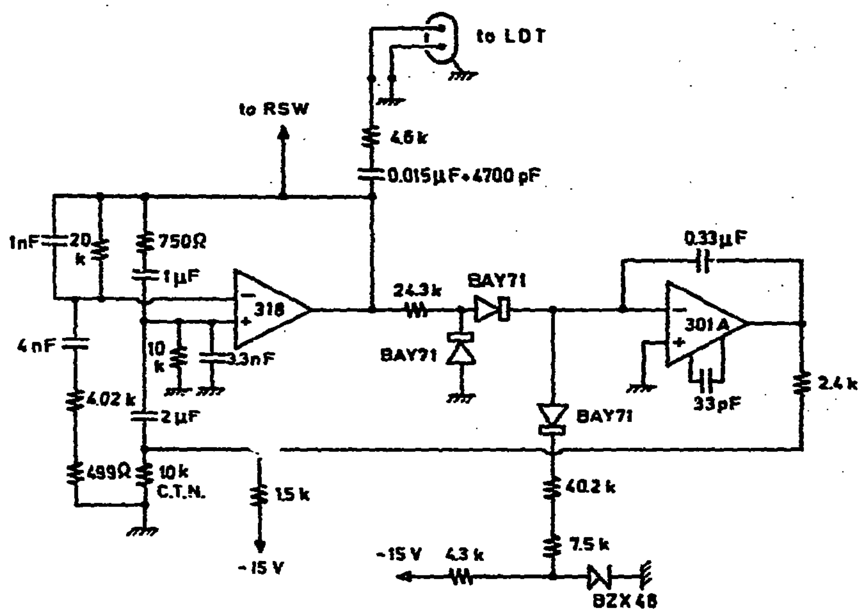
ACKNOWLEDGEMENTS

The authors are indebted to many people at the electronic and instrumentation service, especially: i) to H. Van Trappen and G. De Wilde for guiding students in making the first steps in transposing parts of the vacuum-tube system into semi-conducting circuits; (ii) to H. Vandenhoudt for technical assistance during the build up of the final IC balance control system; and iii) to R. De Clerck who built, in June 1974, an IC version of the data logging system (ref. 3) and adapted it later to the Solartron DVM.

REFERENCES

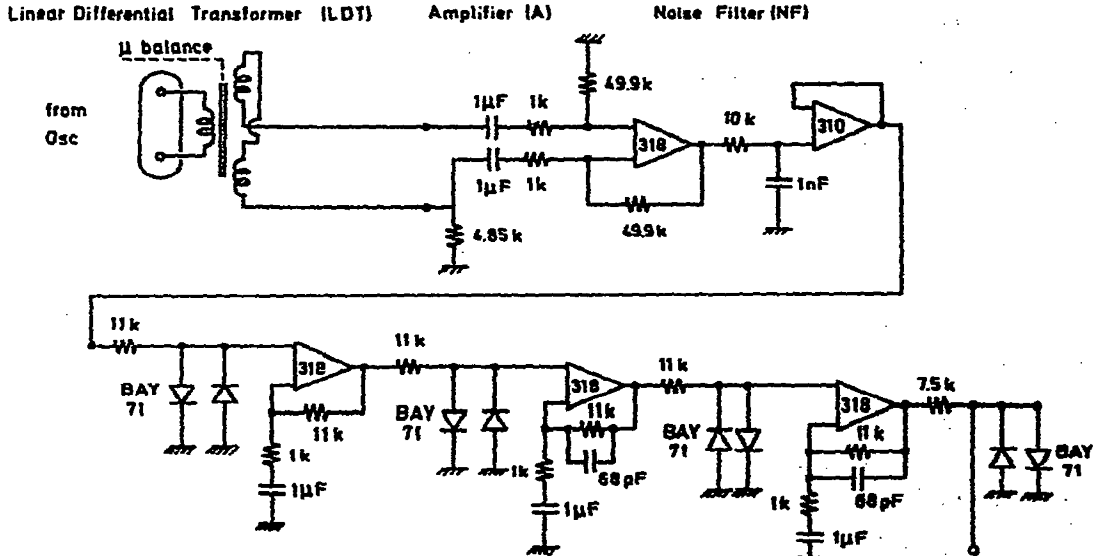
1 A. Van den Bosch, in K.H. Behrndt (Ed.), Vacuum Microbalance Techniques, Vol. 5, Plenum Press, 1966, p. 77.
2 A. Van den Bosch, BLG-190 (1963) S.C.K./C.E.N., B-2400 MOL, Belgium
3 A. Van den Bosch, in C. Eyraud and M. Escoubes (Eds.), Progress in Vacuum Microbalance Techniques, Vol. 3, Publ. Heyden (London), 1975, p. 361.
4 A. Van den Bosch, in C.H. Massen and H.J. Van Beckum (Eds.), Vacuum Microbalance Techniques, Vol. 7, Plenum Press, 1970, p. 9.
5 R.L. Schwoebel, in A.W. Czanderna and S.P. Wolsky (Eds.), Microweighing in Vacuum and Controlled Environments, Elsevier Scientific Publishing Company, 1980, p. 58-93.

Appendix. The Circuits



8kHz Oscillator (Osc)

Fig. A1



Wave shapener (SW)

Fig. A2

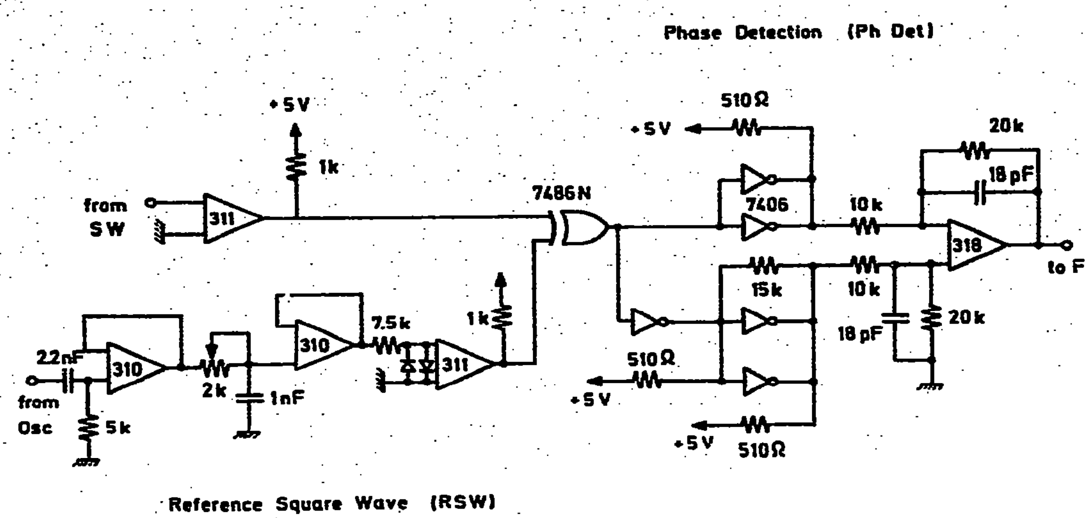

Phase Detection (Ph Det)
+5V
1k
from SW
311
7486N
510Ω
+5V
7406
20k
18 pF
10k
318
to F
15k
10k
18 pF
20k
2.2nF
from Osc
5k
310
2k
1nF
310
7.5k
311
1k
510Ω
+5V
+5V
510Ω
Reference Square Wave (RSW)

Fig. A3

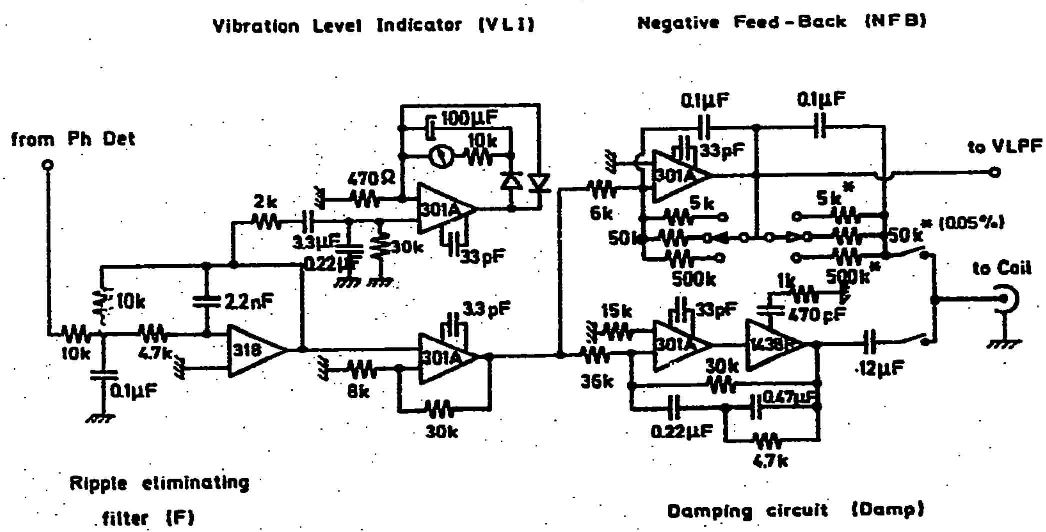
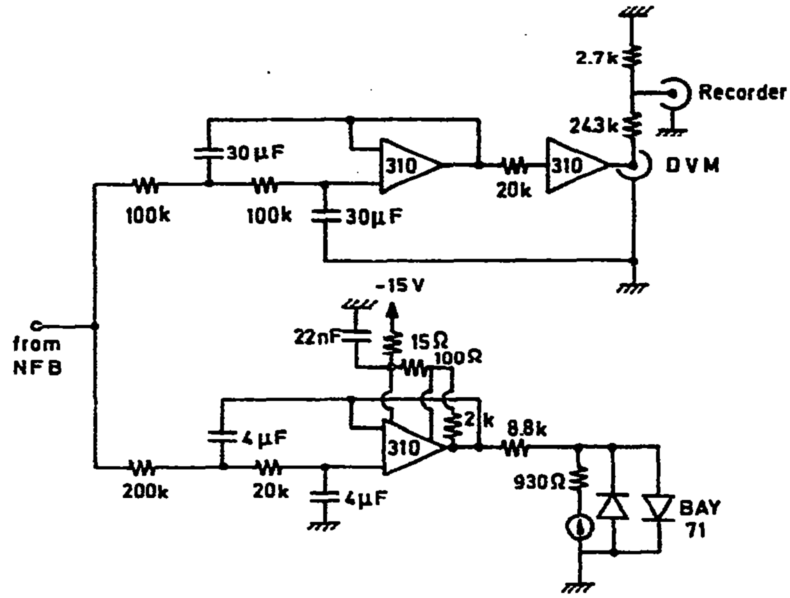
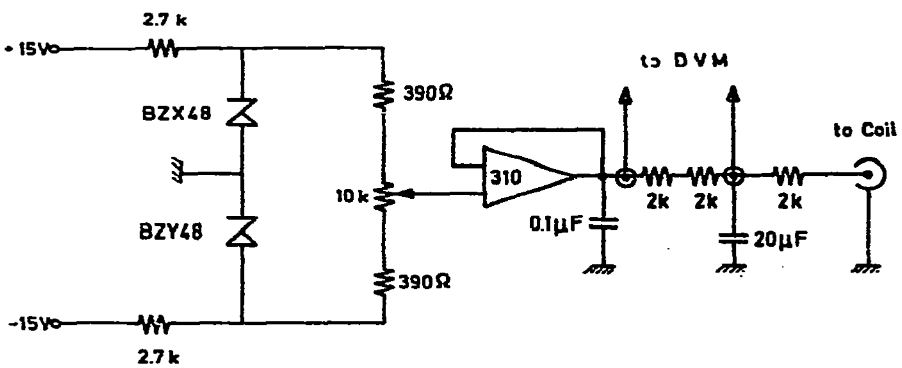

Vibration Level Indicator (VLI)
Negative Feed-Back (NFB)
from Ph Det
100μF
10k
470Ω
2k
301A
3.3μF
0.22μF
30k
33pF
10k
2.2nF
10k
4.7k
318
0.1μF
3.3pF
301A
8k
30k
Ripple eliminating filter (F)
0.1μF
0.1μF
33pF
301A
to VLPF
6k
5k
5k*
50k
50k* (0.05%)
500k
500k*
to Coil
1k
15k
33pF
470pF
301A
1438R
36k
30k
.12μF
0.22μF
0.47μF
4.7k
Damping circuit (Damp)

Fig. A4



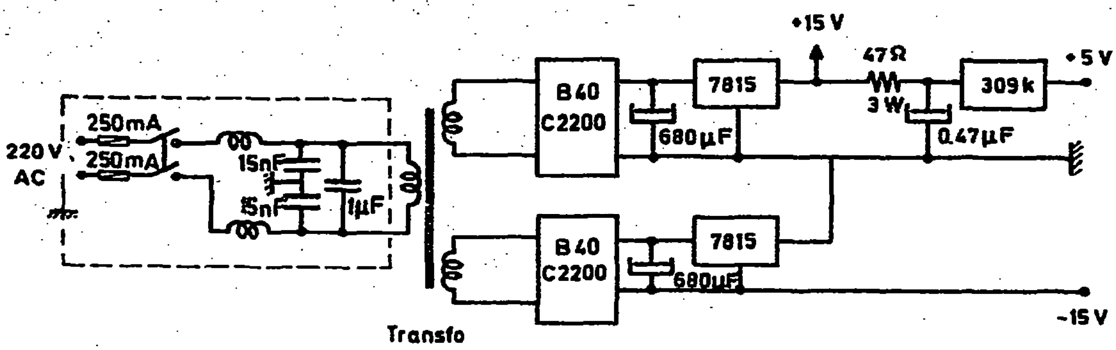
Very Low Pass Filter (VLPF)

Fig. A5



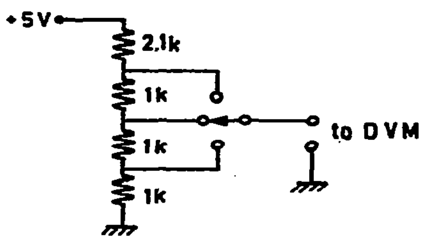
Zero Taring (T)

Fig. A6



Power Supply

Fig. A7



Range Indication

Fig. A8