These comply with stated specifications [signature] 9/25/90

FLUORESCENT X-RAY ION CHAMBER DETECTOR

OPERATIONAL NOTES

As shown in Figs. 1 and 2, the sample is positioned at 45° to the incident x-ray beam within the sample box which can be flushed with He to minimize absorption and scattered radiation. The fluorescent x-rays from the sample pass through a filter (see Table 1) chosen to be absorbing at the absorption edge but that will transmit the fluorescent radiation, i. e. the appropriate atomic number of the absorbing material is Z-1 for the K edge of elements above Ti. After the filter the fluorescent x-rays pass through a slit assembly (1) oriented so as to minimize the passage of secondary fluorescent radiation arising in the filter while maximizing passage of the desired fluorescent radiation into the chamber. In effect the slit assembly makes the filter about 10 times more effective than without it (2). Note that the slit assembly can be unscrewed and rotated 180° and the sample holder fixed in the alternate position in order to reverse the beam direction through the detector. **It is important that the relative positions of the sample holder, slits and x-ray beam are as shown in Fig. 1. The slits point in the same direction as the x-ray beam!** In Table 1 a range of elements, appropriate thicknesses, and their filtering efficiency are listed. Suitable filters are available from EXAFS Materials Co., 871 El Cerro Blvd. Danville, CA 94526, phone (415) 838-7162. The problem of choosing filters for L edge spectroscopy is more difficult but usually can be managed with the K edges of the available elements (2).

ELECTRONICS

The ion chamber consists of a gas tight, 6 μM aluminized Mylar outer window and two inner grids of 90% transparent Ni mesh. The Mylar window and both meshes are captured at their edges between conducting rings separated from each other within the ion chamber body by Teflon insulators. Electrical contact with the rings is made internally through the BNC connectors to either the ground plane (coax 2 and 3) or the amplifier (coax 1). The entrance window and back plane (EO) of the ion chamber are held at -45 V with respect to the center collecting mesh which collects the secondary electrons produced by the fluorescent x-rays. It is held at virtual ground by the inverting input of the Analog Devices 310K Varactor Bridge Operational Amplifier (AD310K). Different gains of the AD310K first stage amplifier can be achieved by changing the feedback resistor R1 (Available from Victoreen, 6000 Cochran Road, Solon, Ohio 44139, phone (216) 248-9300; part number RX-1 Hi Meg Resistor, sizes from 10^6 to $10^{12}\,\Omega$.) In typical ion chambers when delivered R1 = $10^9\,\Omega \pm 5$ %. This special carbon resistor is sealed in glass and has a temperature coefficient of 1000 ppm/^{o}C and may show some long term drift. This may be accommodated with the zero adjust, discussed below. However, the more critical effect of temperature on R1 can be condensation on the glass envelope of the resistor which provides a leakage path which will short out the amplifier.

The output voltage of the AD310K is the input current (amps) times the R1 feedback resistor (ohms), thus 100 pico amps in the ion chamber produces 0.1 volt at the AD310K output when R1 = $10^9\,\Omega$. A low leakage (>$10^{14}\,\Omega$) polystyrene capacitor C1 is wired in parallel with R1 to reduce noise and set the time constant of the device. For R1 = $10^9\,\Omega$ and C1 = 20 pf **the time constant is 0.018 second. This value should be matched with the time constant of the I_0 detector.** There is also a concurrent reduction in bandwidth which is proportional to 1/R1xC1. The time constant and bandwidth may be arbitrarily adjusted by varying C1. The gain of the 310K may be varied only by varying R1. *Early versions of this unit were shipped with R1 = $10^{10}\,\Omega$; however, the more intense beam lines available today often produced instances where the detector was saturated. Consequently, R1 was changed to $10^9\,\Omega$.*

ZERO ADJUST

The AD310K must be balanced by adjusting a 10 turn trimpot (R8) while measuring the voltage at the output of the 310K. This point is identified as "1" on the circuit board. Access must be made by first removing the aluminum cover. The proper balance is achieved when the output is zero when no radiation is entering the ion chamber. The output of the AD310K first stage is coupled to the non-inverting input of the second stage OP27EJ low noise operational amplifier, through R6 and C3 which provide isolation and noise filtering. The gain of this stage (1, 10, 100, 1000) is controlled by switch S1 and the feedback resistor network. Resistors R2, R3, R4 and R5 divide the output and return it to the inverting input of the OP 27EJ. The impedance seen by the inverting input is held constant at 10KΩ by R14, R15, R16, and R17 depending on the gain selected. Since input bias currents flow through equal resistances, the offset voltages produced cancel each other. If this offset is nulled out at the highest gain selected, it will be nulled on all ranges. **The offset is nulled by adjusting trimpot R9** which is identified on the outside of the aluminum cover and is accessible through a hole. The value is measured at the output BNC. Since most analog to digital converters must have a positive voltage, the null should be adjusted to ~ 0.01V. *Note that a sensitive, digital milli-volt meter must be used for these adjustments.*

Capacitor C2 reduces the noise of the second stage without being a significant factor in the bandpass of the overall amplifier, since the time constant of R1C1 is much more restrictive than the time constant RS1C2 (= 2×10^{-4} seconds, worst case). The output of the OP27EJ is protected from external shorts by a 100 Ω resistor (R7) which is connected to the output BNC. Both the amplifiers are powered by an isolated and regulated ± 15V power supply, P1. To minimize noise this power supply is housed externally from the primary circuit board using a twisted pair (TP1) cable. To filter the power supply noise from the AD310K, an RC network composed of two Ta electrolytic capacitors (C4, C6), two ceramic capacitors (C5, C7) and two resistors (R12,R13) are used along with the LC circuit of C8 and L1, L2 (0.7 µH ferrite beads). A less sophisticated power supply filter is used for the OP27EJ which is composed of two ceramic capacitors (C9, C10). Ultimately, the noise of the overall system is limited by the input current noise of the first stage AD310K amplifier. The input current noise specification is ~ 5×10^{-15} amps. Thus, with R1 = 10^9 and a maximum gain of 10^3 for the second stage the approximate low limit amplifier noise is $5 \times 10^{-15} \times 10^9 \times 10^3 =$ ~ 5 mV. This value has been measured using a fast sampling digital volt meter.

The normal operation mode consists of: mounting the sample on the back side of the sample holder, purge the sample chamber with He when operating at the Ti K edge or below--allow purge time after changing the sample, use a very slow Ar flow through the ion chamber with no bubbler in line to minimize microphonic noise generated by motion of the ion chamber window, balance the AD310K using R8 and then obtain some offset (~ 0.01 V) at the BNC output using R9.

For use at high or low x-ray energies use the appropriate fill gas from Table 2. Select one so that the mean free path of the detected x-rays is approximately 1 cm. The absorption path within the ion chamber is ± 1.5 cm from the center collector. Typically use nitrogen below 3 KeV, argon from 3-7 KeV and krypton above 7 KeV. Even at 20 KeV the detector will detect most of the incident x-rays. For economy use low purity krypton and after flushing, valve off the ion chamber to seal it. Diffusion is slow enough so that filling once per shift should be adequate (see Fig. 3). Pinch clamps and flexible tubing work well but ***be careful about the valving sequence or pressure on the fragile window will blow it off!*** The performance of the ion chamber at energies down to 2.4 KeV has been described (3).

IF THE PERFORMANCE OF THE AMPLIFIER IS UNSATISFACTORY FOR ANY REASON, AN EXTERNAL AMPLIFIER (KEITHLEY) MAY BE SUBSTITUTED BY CONNECTING DIRECTLY TO THE CENTER (WHITE) BNC. KEEP THE OTHER TWO BNCS CONNECTED SO THAT PROPER VOLTAGES WILL BE APPLIED TO THE COLLECTING GRIDS IN THE ION CHAMBER.

REFERENCES

1. E. A. Stern and S. M. Heald, Rev. Sci. Instr. **50**, 1579 (1979).
2. F. W. Lytle, "Experimental X-ray Absorption Spectroscopy" in Applications of Synchrotron Radiation, Winick, Xian, Ye and Huang, Editors, Gordon and Breach, pp. 135-223, 1989.
3. F. W. Lytle et al., Nucl. Inst. Meth. **226**, 542 (1984).

Farrel W. Lytle, Sept. 1990
The EXAFS Co.
10815 24th Ave. S.
Seattle, WA 98168 phone (206) 242-9084

PARTS LIST

AD310K	- Analog Devices 310K Amplifier.
B1	- 45 V Battery, Eveready 415 or equivalent
BP1, BP2	- Miniature Spring-loaded Binding Posts (*optional*).
C1	- Polystyrene Capacitor - 20 pf.
C2	- Ceramic Capacitor - 2000 pf.
C3	- Ceramic Capacitor - 22 pf.
C4, C6	- Tantalum Electrolytic Capacitor - 10 uf.
C5, C7, C9, C10	- Ceramic Capacitor - 0.1 uf.
C8	- Ceramic Capacitor - 0.01 uf. - Ceramic Capacitor - 0.1 uf.
COAX 1, COAX 2/3	- Low Noise 50Ω Coaxial Cable (*Amp 21-537*).
EO	- Outer Electrodes - One 6μm Al Mylar, One Ni Mesh Screen.
EC	- Central Electrode - Ni Mesh Screen.
OP27EJ	- National Semiconductor FET Operational Amplifier.
L1, L2	- Ferrite Beads (*0.7 uH*).
P1	- 15 Volt, Isolated, Regulated Power Supply (*Accpian 1505*).
R1	- 10^8Ω to 10^{12}Ω (*typ. 10^9*) Victoreen Carbon Resistor (± 5 %).
R2	- 1% Resistor 90 KΩ
R3	- 1% Resistor 9 KΩ
R4	- 1% Resistor 900Ω
R5	- 1% Resistor 100Ω
R6	- Resistor 10 KΩ
R7, R12, R13	- Resistor 100Ω
R8	- 10 Turn Trimpot 100 KΩ
R9	- 10 Turn Trimpot 5 KΩ
R10, R11	- Resistor 5KΩ
R14	- 1% Resistor 10 KΩ
R15	- 1% Resistor 1 KΩ
R16	- 1% Resistor 9.09 KΩ
R17	- 1% Resistor 10 KΩ
S1	- 4 Position Rotary Switch
TP1	- Twisted Pair Cable

Table 1. FILTERS FOR FLUORESCENT EXAFS DETECTOR

Consider a filter of 3 absorption lengths thickness, i.e., $(\mu/\rho)\ \rho X = 3$, $e^{-3} = .05$.

Thus there would be 95% rejection of the scattered radiation. Two filters [or $6 = (\mu/\rho)\ \rho X$] would give 99.75% rejection.

Element	Density, gm/cm^3	Mass Absorption Coef. Below and Above K-edge μ/ρ, cm^2/gm	Thickness (microns) for 3 Absorption Lengths	Transmission Below and Above Edge $I/I_0(3)$	$I/I_0(6)$
Ti	4.54	93		.665	.445
		685	9.6	.050	.0025
V	6.11	77		.686	.47
		612	8.0	.05	.0025
Cr	7.19	74		.606	.367
		445	9.4	.05	.0025
Mn	7.42	64		.641	.410
		431	9.4	.05	.0025
Fe	7.86	60		.635	.404
		397	9.6	.05	.0025
Co	8.90	54		.677	.459
		416	8.1	.05	.0025
Ni	8.90	47		.614	.378
		290	11.6	.05	.0025
Cu	8.94	41		.644	.415
		280	12.0	.05	.0025
Zn	7.14	36		.680	.462
		280	15.0	.05	.0025
Ga	5.90	31		.669	.447
		231	22.0	.05	.0025
Ge	5.32	30		.651	.424
		210	26.8	.05	.0025

Beginning with Vanadium the correct K-edge filter is that element defined by Z-1. Beginning with Ru the correct K-edge filter is Z-2. For L-edges appropriate K-edges may usually be found for filters.

Table 2. PROPERTIES OF GASES FOR X-RAY DETECTOR
PHOTOELECTRIC ABSORPTION COEF., cm^2/gm
AND
(one absorption length, cm)

Element	He	N	Ne	Ar	Kr	Xe
Density, gm/cm^3	1.78×10^{-4}	1.25×10^{-3}	9.0×10^{-4}	1.78×10^{-3}	3.74×10^{-3}	5.9×10^{-3}
Absorption Edge(s)eV	24.6	401.6	866.9	3202.9	14325.6	34561.4
					1921.0	LI 5452.8
					1727.2	LII 5103.7
Energy, KeV					1674.9	LIII 4782.2
2	6.317 (887)	493.0 (1.62)	1258 (.88)	513.5 (.92)	3870 (.059)	2158 (.079)
3	1.561 (3589)	148.2 (5.40)	405.6 (2.74)	167.4 (3.35)	1359 (.12)	799.0 (.21)
5	.2611 (21459)	30.66 (26.1)	91.06 (12.2)	438.6 (1.28)	348.9 (.77)	654 (.26)
8	.04951 (114000)	6.853 (117)	21.81 (50.9)	120.2 (4.66)	95.92 (2.8)	310.5 (.55)
10	.02241 (250,000)	3.324 (241)	10.90 (102)	63.38 (8.8)	51.26 (5.2)	170.3 (1.0)
15	.00530 (etc.)	.8793 (910)	3.037 (366)	19.14 (29)	115.2 (2.3)	55.96 (3.0)
20	.001909	.3396 (2356)	1.213 (916)	8.01 (70)	54.84 (4.9)	24.97 (6.8)
30	.0004558	.0884 (9050)	.3294 (3374)	2.295 (244)	18.13 (14.7)	7.32 (22)

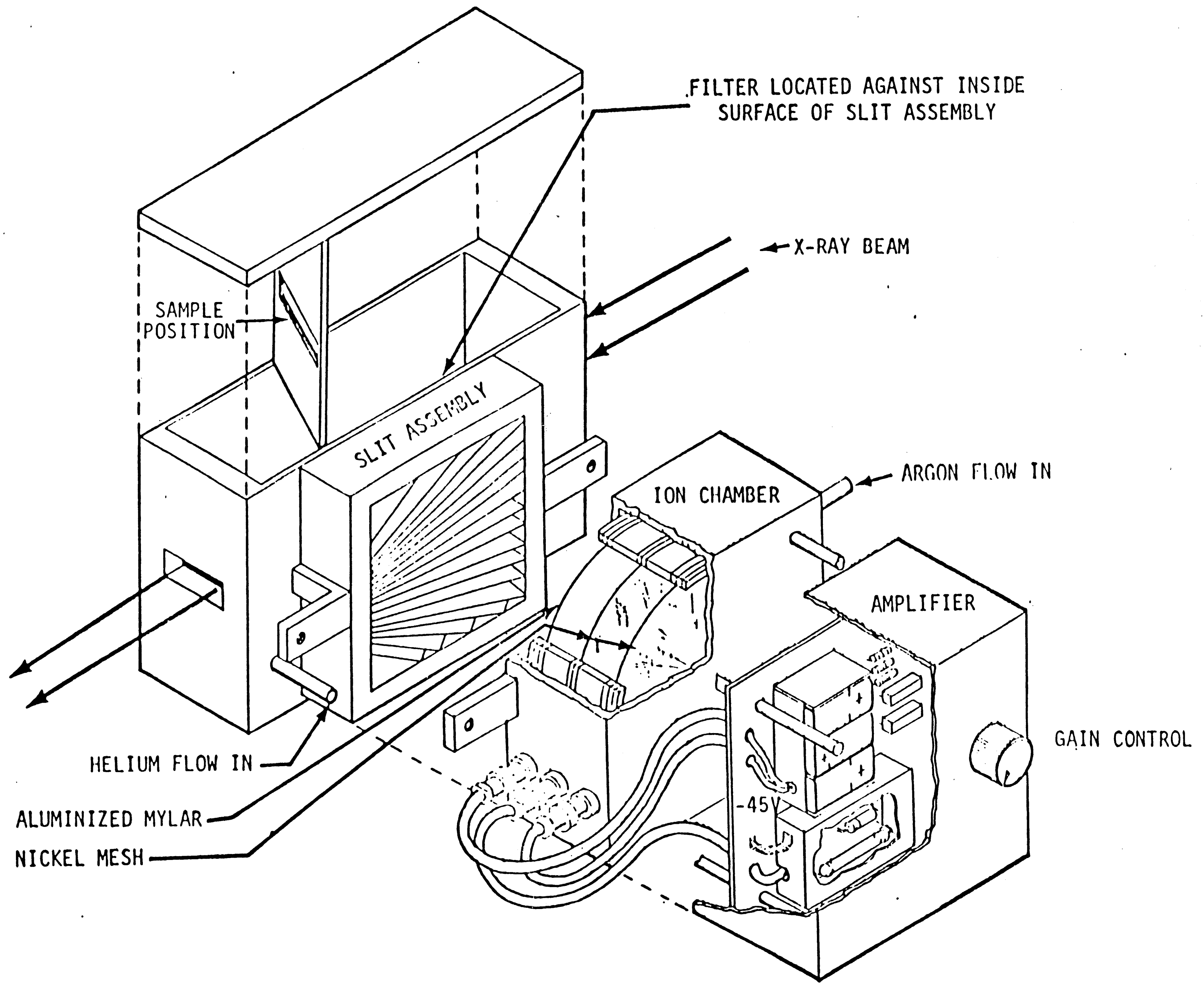

FIG. 1 FLUORESCENT X-RAY DETECTOR

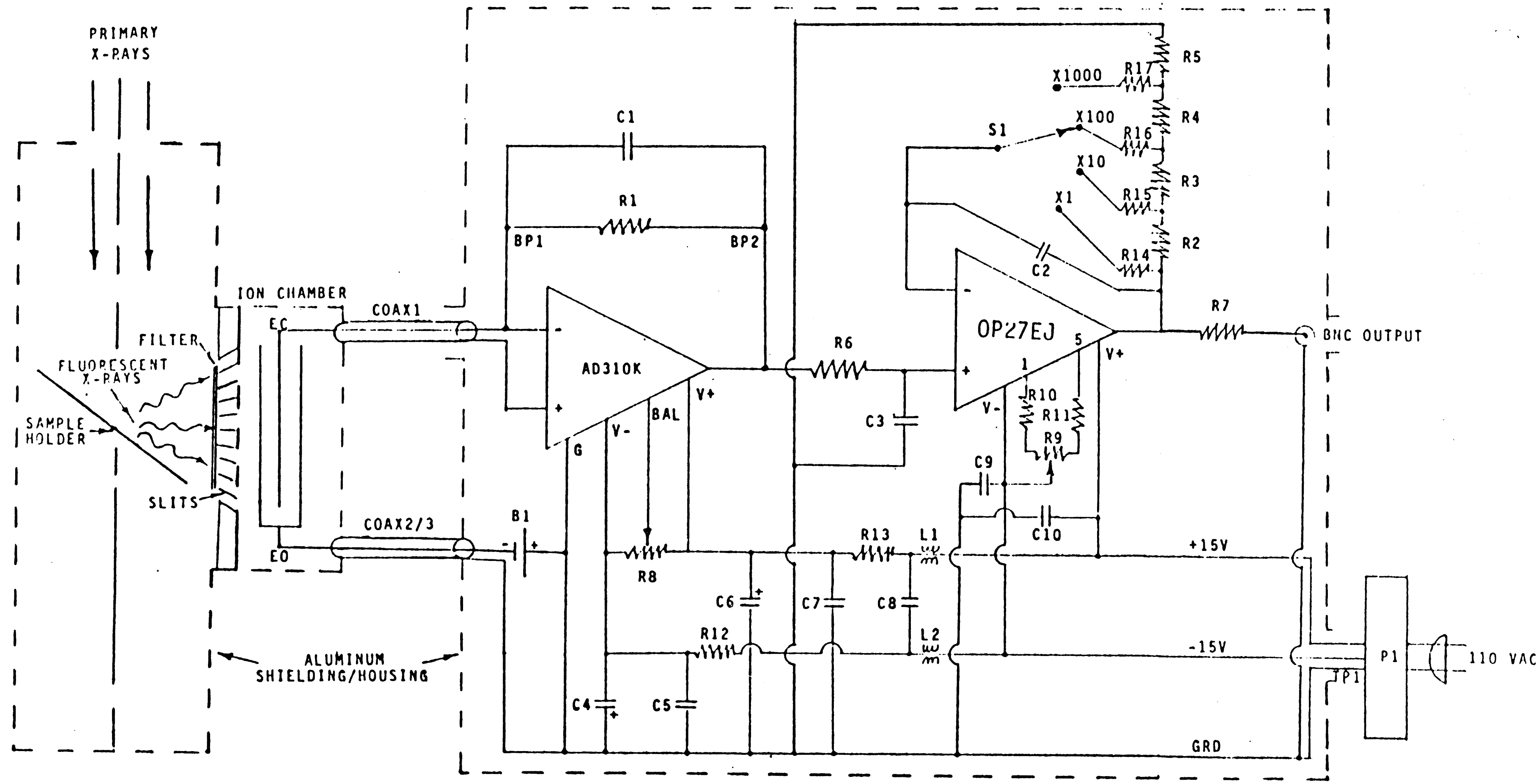

FIG. 2 WIRING DIAGRAM FOR ION CHAMBER AMPLIFIER

Fig. 3.

Performance of Sealed Ion Chamber

In Table 2. note that with argon as a fill gas the ion chamber begins to lose efficiency above 8 KeV. Krypton appears to be the best choice for higher energies. The ion chambers are assembled air tight but there is gradual diffusion through the large, thin, front window. To measure this the chamber was filled with argon and sealed off at T=0 while measuring a ^{55}Fe source (6 KeV). Note that the leakage through the window is slow enough so that filling with a gas and sealing appears to be a good mode of operation, simply flush and refill every few hours. With care a lecture bottle should be sufficient for a few days running. BE CAREFUL NOT TO FLEX THE WINDOW, AS IT LOOSENS THE DETECTOR WILL BECOME NOISY.