STATE OF ALASKA
Department of Natural Resources
DIVISION OF MINES AND GEOLOGY

LABORATORY NOTES NO. 6

Improved Mercury Analysis By XRS

By

Paul L. Anderson

College, Alaska

December 1968

IMPROVED MERCURY ANALYSIS BY XRS

INTRODUCTION

Because of the position of Hg in the periodic table it is necessary to use the L spectrum for analysis. The strongest L mercury line, L_a, occurs at 35.92°2θ (LiF crystal) which is near coincidence with certain tungsten L lines from the primary source, i.e., the WL_{B_1} 37.12°, WL_{B_2} 36.01°, WL_{B_3} 36.55°, WL_L 35.11°, WL_{B_4} 37.72° 2θ. It is not possible to use the HgL_B line at 30.19°2θ because this is interfered with by AsK lines at K_{B_2} = 30.07°, K_{B_5} at 30.19°, K_{B_1} at 30.43°, and K_{B_3} at 30.46° 2θ. A publication by Salmon in 1962 (1) showed that by judicious use of filters the L spectrum from the tungsten primary radiation source could be essentially eliminated. The present investigation uses the method suggested by Salmon.

THEORETICAL

Because As, an element commonly associated with mercury, interferes with the HgL_B line, the HgL_a must be used. Table 1 shows the lines in the region of the HgL_a that may produce sources of interference for practical analyses of mercury.

The most serious problem is with the WL lines. The strongest absorber of WL lines would be an element with an absorption edge just larger than the WL emission lines. The WL emission lines occur at about 1.476°A. Ni has its K absorption edge at 1.488°A and would most strongly absorb the WL lines. A Ni filter placed over the x-ray tube window would absorb both K and LW lines, but would absorb the L about 500 times as great as the WK energy. The result should be that enough K energy would be left to excite the Hg secondary lines, but if the filter thickness is properly chosen most of the WL interference would be absorbed.

EXPERIMENTAL

A one mil thick Ni metal foil of about 1" square was placed over the x-ray tube window. In addition, a one mil thick (1" square) of aluminum was placed over the Ni filter. The Al would help absorb the secondary Ni radiation arising from the filter. Scans were made on stearic acid and on quartz with the filter "sandwich" in place and compared to scans without the filters. The affect of the filter is very striking and is shown in figs. 1 and 2. Practically all the WL_B is absorbed by the one mil of Ni.

Table 1

LINES OCCURING NEAR THE HgL_a ANALYTICAL LINE

ELEMENT	LINE	ORDER	2θ	COMMENT
W	$L_{B_{10}}$	1	35.02	Possible problem, some 2θ separation
W	L_{B_5}	1	35.11	
W	L_{B_7}	1	35.20	
Sb	K_{B_2}	3	35.38	Low intensity, possible problem
W	L_{B_7}	1	35.38	Low intensity, possible problem
Mo	K_{B_4}	2	35.86	Low intensity, possible problem
Mo	K_{B_2}	2	35.92	Low intensity, possible problem
Hg	L_{a_1}	1	35.92	Analytical Hg line
Pb	L_s	1	35.98	Low intensity, possible problem
W	L_{B_2}	1	36.01	Interference
W	$L_{B_{15}}$	1	36.07	Interference
Hg	L_{a_2}	1	36.25	Too low intensity for analytical line
Sb	K_{B_3}	3	36.28	Low intensity, possible problem
Mo	K_{B_1}	2	36.58	Serious interference
Mo	K_{B_3}	2	36.64	Low intensity, possible problem
Sn	K_{B_2}	3	37.00	Very low intensity
W	L_{B_1}	1	37.12	Serious interference
Zn	K_{B_2}	1	37.18	Low intensity, no problem
Zn	K_{B_5}	1	37.21	Low intensity, no problem

There was a small amount of NiK_a that the Al filter did not absorb. These conditions would prevent a Ni analysis when using the filter. A very thin Co filter could possibly be used to absorb all the NiK if that was desired.

It will be noticed that figures 1 and 2 compare the scan with and without the filter, but at different scale factors; $1X10^3$ = full scale with the filter and $5X10^3$ = full scale without the filter. This simply represents the total radiation coming through, and a measure of peak to background is a more practical way of determining the efficiency of the filters. The result of the filter is to remove essentially all the WL_B radiation that interfered with the HgL_a analytical line making the analysis possible.

An analytical line of Hg concentration vs gross counts/second was formed and is shown in fig. 3. Several favorable features are indicated: 1) a straight - line relationship between concentration and x-ray intensity; 2) a detectability of 0.05% Hg; and, 3) an adequate counting rate of about 1000 cps/% Hg.

The effect of the filter on other elements such as Cu, Pb, and Zn was to lower the overall intensity by a factor of about 10, but the peak to background was better with the filter than without. Cu, Pb, and Zn analysis could be run with the filter in place.

Finally, tests were made to see if the other potentially interfering elements shown in table 1, i.e., Sb, Pb, Mo, and Zn, did, in fact, interfere. It was found that 10% of Sb, Pb, and Zn produced no interference, but as little as 0.5% Mo did interfere. One would have to be sure that the Mo in the sample was less than 0.5% for the Hg analysis to be valid.

R E F E R E N C E S

(1) Salmon, Merlyn L.; Practical Application of Filters in X-Ray Spectrography, Advances in X-Ray Analysis; Vol. 6; pp. 301-312; 1962.

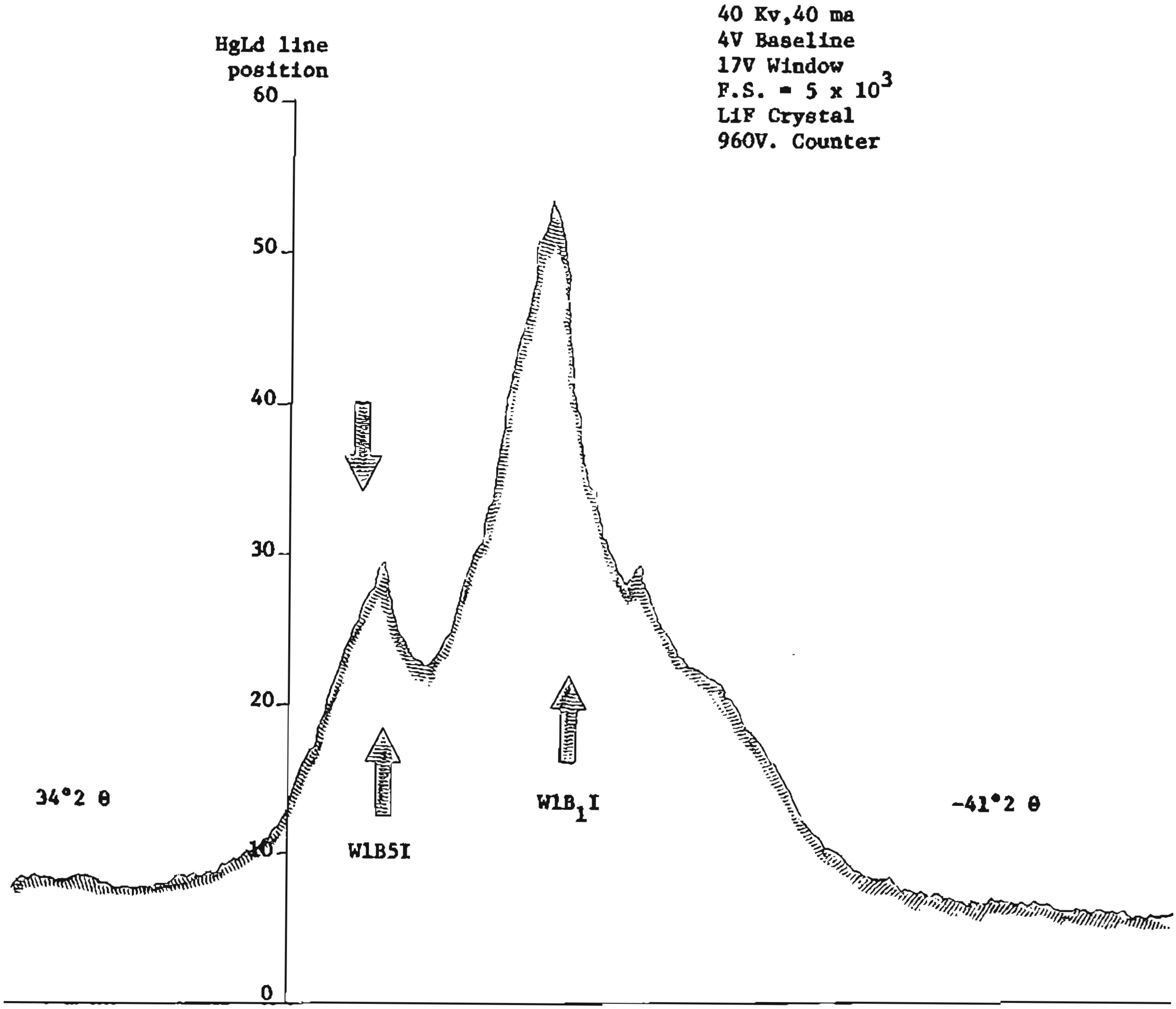

FIGURE 1. Scan from 42° to 34°2 θ with no filter using a quartz scatterer to show the Wl_b lines that interfere with the HgLd analytical line

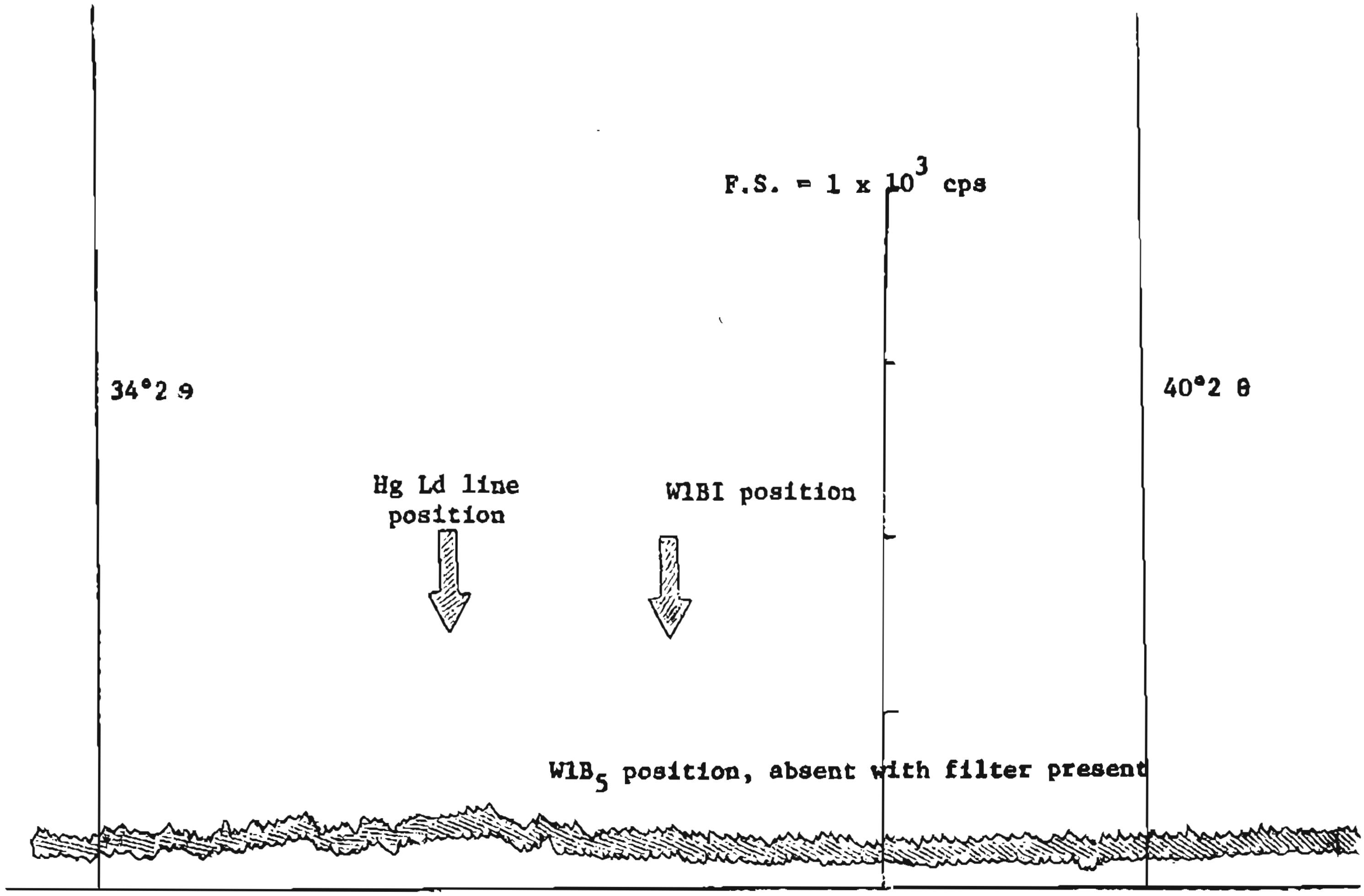

FIGURE 2. Scan from 41° to 34°2θ with 1 mil Ni + 1 Mil Al filters - all other conditions as in Fig. 1; shows no W_b primary radiation.

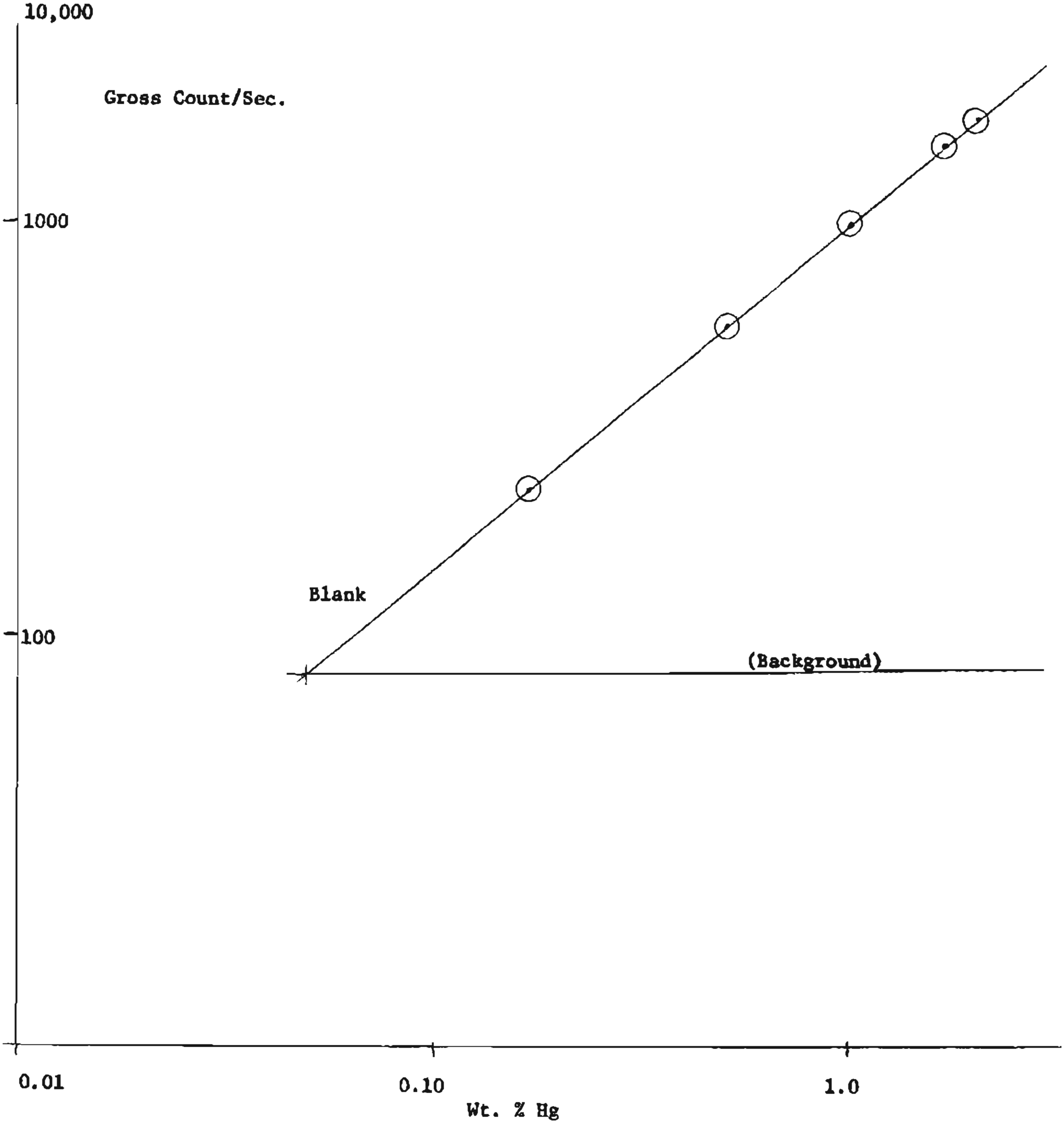

FIGURE 3. Analytical line on HgLd, using filter sandwitch describer.

40 Kv, 40 ma.	LiF crystal
4v Baseline	960 V. Counter
17v Window	Scan counting