

2.3. POWDER AND RELATED TECHNIQUES: X-RAY TECHNIQUES

usually absorbed in the air path or counter-tube window and, hence, are not observed. When using vacuum or helium-path instruments and low-absorbing detector windows, the longer-wavelength fluorescence spectra may appear.

When specimen fluorescence is present, the position of the β filter may have a marked effect on the background. If placed between the X-ray tube and specimen, the filter attenuates a portion of the primary spectrum just below the absorption edges of the elements in the specimen, thereby reducing the intensity of the fluorescence. When placed between the specimen and counter tube, the filter absorbs some of the fluorescence from the specimen. The choice of position will depend on the elements of the X-ray tube target and specimen. If the filter is placed after the specimen, it is advisable to place it close to the specimen to minimize the amount of fluorescence from the filter that reaches the detector. The fluorescence intensity decreases by the inverse-square law. Maximizing the distance between the specimen and detector also reduces the specimen fluorescence intensity detected for the same reason. If the filter is to be placed between the X-ray tube and specimen, the filter should be close to the tube to avoid fluorescence from the filter that might be recorded. It is sometimes useful to place the filter over only a portion of the film in powder cameras to facilitate the identification of the β lines.

If possible, the X-ray tube target element should be chosen so that its β filter also has a high absorption for the specimen X-ray fluorescence. For example, with a Cu target and Cu specimen, the continuum causes a large Cu K fluorescence that is transmitted by an Ni filter; if a Co target is used instead, the Cu K fluorescence is greatly decreased by an Fe $K\beta$ filter. A second filter may be useful in reducing the fluorescence background. For example, with a Ge specimen, the continuum from a Cu target causes strong Ge K fluorescence, which an Ni filter transmits. Addition of a thin Zn filter improves the peak/background ratio (P/B) of the Cu $K\alpha$ with only a small reduction of peak intensity (Ge $K\alpha$, $\lambda = 1.25 \text{ \AA}$; Zn K -absorption edge, $\lambda = 1.28 \text{ \AA}$).

X-ray background is also caused by scattering of the entire primary spectrum with varying efficiency by the specimen. The filter reduces the background by an amount dependent on its absorption characteristics. When using pulse-amplitude discrimination and specimens whose X-ray fluorescence is weak, the remaining observed background is largely due to characteristic line radiation. The β filter then usually reduces the background and the $K\alpha$ radiation by roughly the same amount and P/B is not changed markedly regardless of the position of the filter.

Table 2.3.5.3. Calculated thickness of balanced filters for common target elements

Target material	Filter pair		(A)		(B)	
	(A)	(B)	Thickness mm	g cm ⁻³	Thickness mm	g cm ⁻²
Ag	Pd	Mo	0.0275	0.033	0.039	0.040
Mo	Zr	Sr	0.0392	0.026	0.104	0.027
Mo	Zr	Y	0.0392	0.026	0.063	0.028
Cu	Ni	Co	0.0100	0.0089	0.0108	0.0095
Ni	Co	Fe	0.0094	0.0083	0.0113	0.0089
Co	Fe	Mn	0.0098	0.0077	0.0111	0.0083
Fe	Mn	Cr	0.0095	0.0071	0.0107	0.0077
Cr	V	Ti	0.0097	0.0059	0.0146	0.0066

The β filter is sometimes used instead of black paper or Al foil to screen out visible and ultraviolet light. Filters in the form of pure thin metal foils are available from a number of metal and chemical companies. They should be checked with a bright light source to make certain they are free of pinholes.

The balanced-filter technique uses two filters that have absorption edges just above and just below the $K\alpha_1$, $K\alpha_2$ wavelengths (Ross, 1928; Young, 1963). The difference between intensities of X-ray diffractometer or film recordings made with each filter arises from the band of wavelengths between the absorption edges, which is essential that of the $K\alpha_1$, $K\alpha_2$ wavelengths. The thicknesses of the two filters should be selected so that both have the same absorption for the $K\beta$ wavelength. Table 2.3.5.3 lists the calculated thicknesses of filter pairs for the common target elements. The (A) filter was chosen for a 67% transmission of the incident $K\alpha$ intensity, and only pure metal foils are used. Adjustment of the thickness is facilitated if the foil is mounted in a rotatable holder so that the ray-path thickness can be varied by changing the inclination of the foil to the beam.

Although the two filters can be experimentally adjusted to give the same $K\beta$ intensities, they are not exactly balanced at other wavelengths. The use of pulse-amplitude discrimination to remove most of the continuous radiation is desirable to reduce this effect. The limitations of the method are (a) the difficulties in adjusting the balance of the filters, (b) the band-pass is much wider than that of a crystal monochromator, and (c) it requires two sets of data, one of which has low intensity and consequently poor counting statistics.