The author wishes to thank Dr G. A. Geach for his encouragement during the work; Dr J. Adam of the Atomic Energy Research Establishment, Harwell, for helpful discussions; and Dr T. E. Allibone, F.R.S., for permission to publish this note.

References

Buzzard, R. W., Liss, R. B. & Fickle, D. P. (1952). U.S. Atomic Energy Commission Document No. 3418.
Kiessling, R. (1950). *Acta chem. scand.* **4**, 209.
Laves, F. (1943). *Naturwissenschaften*, **31**, 145.
Makarov, E. S. (1949). *Dokl. Akad. Nauk, S.S.S.R.* **68**, 509.
Nelson, J. B. & Riley, D. P. (1945). *Proc. Phys. Soc.* **57**, 160.
Nowotny, H., Bauer, E., Stempfel, A. & Bittner, H. (1952). *Mh. Chem.* **83**, 221.
Nowotny, H., Stempfel, A. & Bittner, H. (1952). *Mh. Chem.* **83**, 287.
Rundle, R. E. & Wilson, A. S. (1949). *Acta Cryst.* **2**, 148.
Zachariasen, W. H. (1949). *Acta Cryst.* **2**, 94.

Acta Cryst. (1954). **7**, 458

The preparation of a $K\beta$ filter for X-ray crystallography with special reference to chromium radiation.

By E. Krogh Andersen and C. E. Schäffer, *Chemical Laboratory of the Royal Veterinary and Agricultural College, Copenhagen, Denmark*

(*Received* 25 *February* 1954)

The ideal $K\beta$ filter consists of the pure element rolled to a thin foil (Ross, 1928). Where the element is not ductile (or not available) alternative methods have been adopted: (1) A compound of the filtering element with others of low atomic number is spread on a piece of paper coated with shellac, or suspended in a paraffin block, which is afterwards cut to the calculated thickness (Pierce, 1931). (2) Filter paper is soaked in a solution of a compound of the filtering element, and dried. (3) The element is electro-deposited on aluminium foil (Wood, 1931). (4) The compound is mixed with beeswax and pressed between two rocksalt cleavage surfaces at 50° C. The sodium chloride is then removed by dissolution in water (Kratky, 1943). (5) The compound is kneaded uniformly into high-melting-point paraffin wax, and pressed between spaced glass plates (Kirkpatrick, 1942). (6) The filter substance is suspended in collodion, spread out in a thin sheet, and allowed to dry (Sidhu, 1937*a*, *b*).

This last method has been developed here in order to obviate the use of expensive commercial vanadium pentoxide filters. The fact that we have reduced the amount of supporting material used without losing strength and flexibility of the filters, gives them particular value where fourth-period elements (e.g. Ti, V, Cr, Mn and perhaps Co) are involved.

Vanadium tetroxide was prepared by heating pure ammonium metavanadate (analysed for heavy metals) in a covered platinum crucible, using a Bunsen burner. A blue-black product was obtained consisting of vanadium tetroxide (80–90%) and vanadium trioxide (10–20%), the actual composition depending on the time of heating. Prolonged heating favoured the formation of the tetroxide or even the pentoxide. The pentoxide-free product, obtained by strong heating for half an hour, was used here. The following recipe is based on several experiments: For a filter of 24 cm.^{2} area, 0·37 g. 'V_2O_4' was employed, i.e. 5% more than the amount corresponding to 0·009 g. vanadium per cm.^{2}. Prolonged dry grinding in an agate mortar followed by grinding after addition of ethyl acetate eliminated granular matter. 24 drops (i.e. 0·45 g. containing 0·13 g. dry matter) of ethyl acetate–pyroxylin lacquer and half a drop of dibutyl phthalate were added, the latter to make the film flexible. The consistency was made that of a thin gruel by further addition of ethyl acetate, and the mixture was then poured on to a glass plate of area 24 cm.^{2}, spread evenly with a paint brush, and dried in the air. After one night it was possible to strip off the filter with a wet razor blade. It was strengthened by pasting on to it a sheet of condenser paper (weighing 1 mg.cm.$^{-2}$). Application of larger amounts of dibutyl phthalate was unnecessary and impeded the drying. An analysis of the filters indicated that they tended to contain too much pyroxylin near the edge of the glass plate; therefore only the central portion of the plate was used.

In order to compare the different $K\beta$ filters for chromium radiation, the increase in exposure time and the factor reducing the intensity ratio, I_β/I_α, relative to no filters, have been calculated (Table 1). Four different filters have been considered: the metal foil, our 'V_2O_4' filter, an ammonium metavanadate filter (as recommended by Sidhu (1937*a*, *b*)), and the commercial filter. The metavanadate filter contains the same percentage of pyroxylin as ours (i.e. $< 40\%$) while the commercial filter contains $> 80\%$ pyroxylin. Furthermore, the commercial product had only 5·5 mg. vanadium per cm.^{2}.

Table 1. *Comparison of filters*

	Prolongation factor	Reduction factor on I_β/I_α
No filter	1	1
Vanadium foil	2·0	1/53
'V_2O_4' filter	3·0	1/47
NH_4VO_3 filter	4·2	1/44
Commercial filter	6·6	1/11

References

Kersten, H. & Maas, J. (1933). *Rev. Sci. Instrum.* **4**, 14.
Kirkpatrick, P. (1942). *Phys. Rev.* **62**, 302.
Kratky, O. (1943). *Naturwissenschaften*, **31**, 325.
Pierce, W. C. (1931). *Phys. Rev.* **38**, 1409.
Ross, P. A. (1928). *J. Opt. Soc. Amer.* **16**, 433.
Sidhu, S. S. (1937*a*). *Rev. Sci. Instrum.* **8**, 308.
Sidhu, S. S. (1937*b*). *Indian J. Phys.* **11**, 337.
Wood, W. A. (1931). *Proc. Phys. Soc., Lond.* **43**, 275.