the value of bound carbon was higher the weak lines corresponding to Mulford's data reported in Table 1 appeared relatively stronger.

Further evidence of another new carbide phase was obtained on heating plutonium dioxide with carbon sufficient to produce PuC up to 1800 °C, mixing the product with excess carbon and finally heating it to 2400–2600 °C. Out of seven preparations, two identical patterns have been obtained which showed it to be mixture of carbon and the new phase. As the number of lines obtained was not great, no crystallographic deductions are suggested. The diffraction lines of this phase, the spacings of which are quite different from those reported in Table 1, was also detected in the preparations of higher carbides when PuO_2 and carbon were heated first to 1700 °C to 1800 °C for some time and then heated to 2300 °C to 2600 °C.

The X-ray diffraction patterns of all the preparations, loaded in silica capillaries of 0·20 to 0·28 mm internal bore, were taken in a 19 cm Unicam camera with copper radiation. An optical pyrometer was used for measuring the temperatures of the preparations.

The authors express their thanks to Dr H. D. Sharma and Dr A. S. Ghosh Mazumdar for their interest and helpful discussions, to Dr G. A. Welch for initiating the problem and encouragement and to Mr A. V. Hariharan for preparing a few samples carbides in the initial stage of the investigation.

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A special application of the crystal setter.

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In a note on the setting of crystals in X-ray diffraction work one of us described a method and a corresponding instrument for setting crystals (Kulpe, 1963). This instrument ('crystal setter') has been under trial for nearly two years in our Institute. It has proved fully satisfactory and is now used for all our routine setting. The setting procedure is simple, quick, and sufficiently accurate even for relatively big corrections (15° on each of two crossed arcs of the goniometer head).

In favourable cases it may be possible to obtain a set of lattice parameters starting from a single oscillation photograph, without the necessity of using more than one crystal or remounting the crystal on the goniometer head, by setting the crystal corresponding to three non-coplanar directions.

The 'layer lines' corresponding to such directions will in general not be recognizable without the help of the crystal setter. The recognizability is of course better if one of the lattice parameters is fairly large, but then adjustment in a direction corresponding to such a large translation period will be rather difficult. In the following an example of this kind is shown to demonstrate the possibility of obtaining three settings from one oscillation photograph (Fig. 1) and in order to indicate the accuracy obtainable in one or in two steps, respectively. A mica crystal was used.

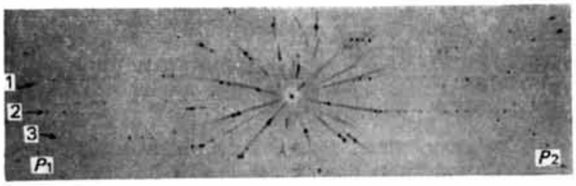

Fig. 1. 10° oscillation photograph of a mica crystal. The arrows 1,2,3 indicate rows of reflexions which may be adjusted as mis-set zero layer lines. P_1, P_2 are marking points.

In Fig. 1 P_1 and P_2 are marks which make it easy to put the film around the crystal setter in the correct position. The three arrows marked 1,2,3 indicate rows of reflexions which were then adjusted as equatorial layers. The photographs were taken with the plane of the lower arc of the goniometer head perpendicular to the X-ray beam. Copper radiation was used.

For the three marked rows the following corrections were read off from the arcs of the crystal setter:

1. $\alpha_\perp = 14,6°$ rhs 2. $\alpha_\perp = 6,3°$ lhs 3. $\alpha_\perp > 15,0°$ lhs
$\alpha_\parallel = 1,8°$ afc $\alpha_\parallel = 7,5°$ tc $\alpha_\parallel = 2,6°$ tc

(rhs = to the right hand side; lhs = to the left hand side afc = away from the collimator; tc = towards the collimator). In the third case the setting $\alpha_\perp$ exeeded the measuring range of the arc (±15°). A second setting photograph enabled $\alpha_\perp$ to be determined to $\alpha_\perp = 16,6°$.

Figs. 2, 3, 4 show oscillation photographs obtained after the corresponding settings. The translation periods along

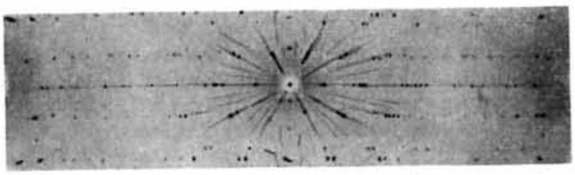

Fig. 2. Oscillation photograph according to case 1 (oscillation angle 10°).

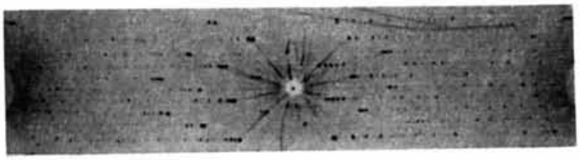

Fig. 3. Oscillation photograph after setting according to case 2: Ni filter, oscillation angle 90°.

the three directions used as axes of rotation are about 5·1 Å; 13·6 Å; 9·6 Å; respectively.

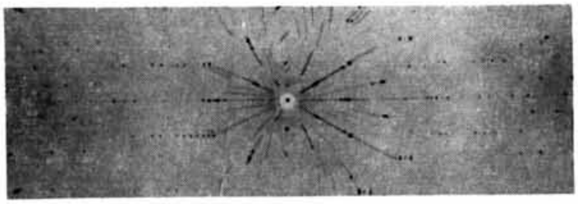

Fig. 4. Oscillation photograph after setting according to case 3; oscillation angle 10°.

Relatively large oscillation angles of 10° (Figs. 1, 2 and 4) or 90° (Fig. 3) were used in order to make it easier to recognize the layer lines. In actual practice smaller oscillation angles suffice.

Experience with the crystal setter shows that in most cases the setting is satisfactory after the first step of adjustment even for relatively large angles of mis-setting. The limit of accuracy in setting a crystal with this instrument lies within the accuracy obtainable in adjusting the goniometer arcs. In a new design of the instrument the measuring range of the arcs has been increased to ± 24°.

No sign conventions are required.

Note added in proof.– Laue photographs taken with a film cylinder corresponding to the transparent cylinder of the crystal setter may equally well be used for setting crystals with the help of the setter.

We should like to thank Mr. G. Schulz for his help in the experimental work.

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X-ray absorption coefficients for certain metals.

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The currently available experimental and theoretical values of X-ray mass absorption coefficients have been discussed in some detail in *International Tables for X-ray Crystallography* (1962). It may be concluded that the reliability of experimental values is somewhat doubtful, even in the limited number of cases when a small experimental error is claimed. Empirical methods of calculating absorption coefficients based on the quantized structure of the atoms have been given by several workers (*e.g.* Victoreen, 1949), but the accuracy of these is not better than a few per cent. In particular the parameters involved are chosen to obtain the best overall fit to certain experimental data and so the reliability of the calculated coefficients will depend on any systematic errors in the experimental data.

The determination of accurate X-ray structure factors on an absolute scale is in many cases dependent on the knowledge of reliable values of the absorption coefficients concerned (see *e.g.* Cooper, 1962). For this reason a programme has been carried out by the author to determine accurate values of the mass absorption coefficients (μ_m) for aluminum, copper and certain first order transition metals, for several commonly used characteristic radiations.

Attenuation constants were determined for uniform foils of the metals with monochromatized X-ray beams, and the mass per unit area of each foil was determined by weighing a disc punched from it. The values of μ_m obtained are given in Table 1 together with their experimental accuracy. The value given for chromium and Ag $K\alpha$ radiation is that determined previously (Copper, 1962).

It is to be expected that log μ_a† is a linear function of log Z with a slope of value close to 4. Plots of log μ_a against log Z for the metals from vanadium to copper showed that such straight lines could be chosen to fit almost all the experimental points within the experimental accuracy given. These lines were therefore used to determine certain values of μ_m not determined experimentally. These values are shown in Table 1 in brackets and it is to be expected that the reliability of these will not be much different from that of the associated experimental values, and that they may therefore prove to be useful.

Table 1. *X-ray mass absorption coefficients for certain metals* (cm^2g^{-1})

	Ag $K\alpha$	Mo $K\alpha$	Cu $K\alpha$
Al	2·65 ± 0·01	5·10 ± 0·02	50·40 ± 0·57
Ti	(11·53)	(22·23)	(187·5)
V	13·71 ± 0·08	25·18 ± 0·14	213·9 ± 3·8
Cr	15·18 ± 0·15	(28·84)	(246·6)
Mn	(16·67)	(32·14)	(274·2)
Fe	19·38 ± 0·10	37·61 ± 0·33	314·3 ± 6·9
Co	21·14 ± 0·11	40·40 ± 0·32	(350·0)
Ni	24·45 ± 0·13	46·41 ± 0·36	48·96 ± 0·41
Cu	25·64 ± 0·14	48·88 ± 0·78	51·84 ± 0·43
	Co $K\alpha$	Fe $K\alpha$	Cr $K\alpha$
Al	76·19 ± 0·85	96·8 ± 1·2	156·0 ± 3·7
Ti			
V	315·6 ± 5·7	387·7 ± 7·0	82·7 ± 2·8
Cr			(88·7)
Mn		(61·1)	(99·1)
Fe	56·45 ± 0·49	69·7 ± 1·1	112·2 ± 1·8
Co	61·56 ± 0·50	78·0 ± 0·6	(126·0)
Ni	72·8 ± 1·2	91·5 ± 1·5	146·5 ± 2·3
Cu	77·2 ± 1·2	96·0 ± 1·5	154·5 ± 2·5

Whilst great care was taken in the present study to avoid systematic errors it is well known that accurate absorption coefficients measured by different workers may vary quite appreciably. It is to be hoped therefore that further determinations may be made by other workers in order to provide a more realistic indication of the reliability of such measurements.

I should like to thank Professor Sir Nevill Mott, F.R.S. and Dr W. H. Taylor for provision of facilities and for their interest and encouragement. I am also indebted to Mr Roger Morley for carrying out many of the weighings.

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† The notation is that used in *International Tables* (1962).