Acta Cryst. (1968). A**24**, 699

On the application of Zachariasen's extinction correction to a set of visually estimated intensity data.

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(*Received* 24 *April* 1968)

Zachariasen's extinction correction has been applied to a set of visually estimated intensity data. Least-squares refinement showed that a fairly drastic removal of all strongest reflections (~10% of the data) gave a somewhat better result than applying the correction.

In a recent refinement of the structure of MoO_2 (Magnéli, 1946; Brandt & Skapski, 1967) we found that our visually estimated intensity data suffered quite obviously from extinction effects. The details of the refinement were published when R was 0·053 after 35 strong reflexions (out of a total of 1379) had been removed selectively. The low R index encouraged us to try and see whether the application of the extinction correction according to Zachariasen (1963) would improve the result significantly.

The following sequence of refinements was carried out in space group $P2_1/c$ with a full-matrix least-squares program. (*a*) A refinement using all the data. (*b*) Refinements in which increasing numbers of reflexions above a certain limit were removed. When a total of 139 reflexions were removed there was no further decrease in R and the standard deviations were at a minimum. (*c*) On the basis of this refinement, Zachariasen's extinction correction was applied to all reflexions, with the use of the absorption program of Coppens, Leiserowitz & Rabinovich (1965) modified by S. Åsbrink and B. G. Brandt. The results are summarized in Table 1.

As can be seen, the extinction correction produces a significant improvement both in the R value and in standard deviations. Nevertheless we find that a fairly drastic removal of the strongest reflexions (in this case ~10% of the data) gives a slightly better result. As this procedure is in any case an intermediate step in applying the correction we feel that for visually estimated data it might be better to stop at this point. The parameters obtained in refinements (*b*) and (*c*) are very similar and are not significantly different from those published previously by us.

The extinction correction has been applied to diffractometer data by both Zachariasen (1963) and Åsbrink & Werner (1966). In the latter paper, however, the authors specifically examine the relative merits of applying the correction and of omitting a sizable number of the strongest reflexions. They were able to show that for their diffractometer data slightly better results are obtained by applying the correction. In diffractometer and possibly photometrically estimated data, however, the strongest reflexions tend to be the most accurately measured. This is not usually the case with visual data.

This work has been supported in part by the Swedish Natural Science Research Council.

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Table 1. *Summary of results of successive refinements*

(*a*) All reflexions; $R=0·071$.
(*b*) 139 strongest reflexions removed; $R=0·049$.
(*c*) All reflexions extinction corrected; $R=0·052$.

		$x\pm\sigma(x)$*	$y\pm\sigma(y)$	$z\pm\sigma(z)$	$B\pm\sigma(B)$
Mo	(*a*)	0·23164 ± 10	0·99118 ± 14	0·01647 ± 10	0·062 ± 6
	(*b*)	0·23167 ± 5	0·99156 ± 7	0·01646 ± 5	0·181 ± 4
	(*c*)	0·23167 ± 6	0·99176 ± 8	0·01644 ± 6	0·184 ± 4
O(1)	(*a*)	0·1130 ± 12	0·2188 ± 13	0·2339 ± 12	0·30 ± 5
	(*b*)	0·1122 ± 6	0·2173 ± 7	0·2332 ± 6	0·38 ± 2
	(*c*)	0·1119 ± 7	0·2170 ± 7	0·2332 ± 7	0·37 ± 3
O(2)	(*a*)	0·3905 ± 12	0·6984 ± 13	0·2981 ± 12	0·30 ± 4
	(*b*)	0·3907 ± 6	0·6964 ± 6	0·2988 ± 6	0·36 ± 2
	(*c*)	0·3905 ± 7	0·6968 ± 7	0·2986 ± 7	0·35 ± 2

* The previously published standard deviations were obtained with a block-diagonal least-squares program, so that no direct comparison can be made with the figures in this Table.