So dead time can cause errors in both $N_m$ and $R_m$ which compound to yield $N_m R_m$ rather than $N_i R_i$. The full range of error will be almost twice that indicated in Fig. 1, owing simply to lost counts. The actual specific error magnitude will depend upon the count rate at which the attenuator ratio was established originally.

The form of this error is a shortfall which becomes progressively greater with increasing intensity, a trend akin to that associated with extinction. In practice, the two effects would not be readily distinguished and that due to dead time would simply be absorbed as an extinction effect. The resultant extinction parameters would not then accord with physically realistic values.

Incorporation in the data collection procedure of the appropriate correction, derived by Chipman's procedure and applied as in (1), would eliminate error due to dead time, establish constant attenuation ratios and ensure that $N_i R_i$ was obtained. This would assist in isolating those effects strictly due to extinction.

While correction for dead time allows for linearity of counting to higher count rates, it would be ill-advised to push this too far, say beyond 10 15 Kcounts s$^{-1}$, since resolving capability could be degraded, *vide* Mathieson (1982).

The purpose of this note is to draw attention to a source of error in standard procedures for measuring intensities on a diffractometer and thus to aid in improving the accuracy of the resultant structure factors.

References


**Precise lattice parameter determination of PtHg$_4$.** By S. K. LAHIRI, J. ANGILELLO and M. NATAN, IBM

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Abstract

The lattice parameter of cubic PtHg$_4$ at 298 K is 6.2001 Å and it is accurate to better than 1 part in 10 000.

Interest in the Pt–Hg contact reaction has increased recently because of the use of such contacts in experimental Josephson device packages (Anacker, 1979; Lahiri, Geldermans, Kolb, Sokolowski & Palmer, 1980). The kinetics of the reaction and morphology of the reaction product have been studied recently in detail (Lahiri & Gupta, 1980; Barlow & Planting, 1969). In the presence of excess Hg, reaction at temperatures between 293 and 426 K results in the formation of primarily PtHg$_4$ on the Pt surface. The lattice parameter of the overlying PtHg$_4$ has been found to be equal to 6.2047 Å. However, the magnitude of the strain present in this PtHg$_4$ layer could not be determined with reasonable accuracy, mainly because of the fact that the published (Bauer, Nowotny & Stempfl, 1953) lattice-parameter value of 6.18 Å for bulk PtHg$_4$ is not considered precise enough. The present work was thus undertaken for the determination of the bulk PtHg$_4$ lattice parameter with greater precision with an X-ray diffraction technique.

The PtHg$_4$ samples were prepared by reacting 0.025 mm thick Pt foils of 99.97% purity with triple-distilled Hg (Pt weight:Hg weight $\approx$ 1:10) in evacuated and sealed pyrex capsules. The reaction was carried out by annealing the capsule at 423 K for 11 d, a period which is sufficient to convert all of the Pt into PtHg$_4$ (Lahiri & Gupta, 1980). The Pt foils were found to disintegrate upon completion of the reaction resulting in the formation of fine PtHg$_4$ particles dispersed in liquid Hg. After the reaction the capsule was broken; the particles were allowed to settle and part of the sediment was smeared over a glass slide for X-ray diffraction study. The diffraction sample thus contained a small amount of Hg in addition to the embedded particles of PtHg$_4$, a compound which has been reported (Jangg & Dortbudak, 1973) to be stable only over a very narrow composition range.

Diffraction patterns were obtained with copper radiation and an IBM S 7 computer-controlled Norelco vertical diffractometer equipped with a post-beam monochromator. The diffraction data were transferred afterwards into an IBM 370 computer for processing and analysis. With a...
method published earlier (Segmuller, 1970), each peak position was identified with the angle that corresponded to a change from a positive to a negative slope. The angle was obtained through computation and extrapolation of the derivatives of the line profile at various points on both sides of the peak.

The peak positions, interplanar spacings and relative intensities obtained for all reflections by analyzing the PtHg₄ diffraction pattern are shown in Table 1. For precise determination of the PtHg₄ lattice parameter \(a_0\), the lattice parameter values calculated for the high-angle reflections \((2\theta > 70°)\) are plotted in Fig. 1 as a function of the Nelson-Riley-Sinclair-Taylor parameter (Barrett & Massalski, 1966), \(0.5 \frac{\cos^2\theta}{\sin \theta} + \cos \theta/\theta\). The \(a_0\) is then given by the intercept of the straight line drawn through the data points using the least-squares method. The value of the lattice parameter, thus obtained, is equal to \(6.2001 \pm 0.0007\) Å.

Use of an alternative parameter, \(\frac{\cos \theta}{\sin \theta} + \cos^2 \theta/\theta\), for extrapolation changes the lattice parameter to \(6.20010 \pm 0.00016\) Å. This value compares excellently with the published values (Donnay & Ondík, 1973; Barrett & Massalski, 1966) of 6.18 Å.

In order to determine the accuracy of our measurement, the lattice parameter of a standard Au powder sample was also determined at 296 K with the same technique. The lattice-parameter values determined for this sample from different reflections are also plotted in Fig. 1. From this plot the lattice parameter of Au is found to be equal at 4.07886 \(\pm 0.00016\) Å. This value compares excellently with the published values (Donnay & Ondík, 1973; Barrett & Massalski, 1966) of 4.07879 and 4.0788 Å at 298 K (corresponding to 4.07886 and 4.0787 Å, respectively, at 296 K) indicating that the accuracy of our measurement technique is better than 1 in 10 000.

The reported lattice parameter of the PtHg₄ layer that forms on bulk Pt upon exposure to Hg (Lahiri & Gupta, 1980) is \(~ 0.046\) Å larger than that measured for bulk PtHg₄ in the present work. Accordingly, the strain in the former appears to be much smaller \((~ 7 \times 10^{-4})\) than the strain \((~ 4 \times 10^{-3})\) that one would have obtained with the previously reported lattice parameter of 6.18 Å.

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References


