SHORT COMMUNICATION

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.


X-ray powder diagrams predicted for certain even-n alkanes. By S. C. Nyburg and F. H. Pickard, Lash Miller Chemical Laboratories University of Toronto, Toronto, Ontario, Canada M5S 1A1

(Received 6 February 1974; accepted 11 March 1974)

From the known crystal structure of an n-alkane it is possible to predict the structures of all those n-alkanes isostructural with it. These predictions are matched against published X-ray powder data for n=10, 12, 14 and 16. The overall agreement is good but some discrepancies, notably some predicted moderately strong reflexions not apparently observed, are not readily explained.

Norman & Mathisen (1972), hereinafter N & M, have published a note on the X-ray powder diagrams and derived cell dimensions for the triclinic n-alkanes having n=10, 12, 14 and 16.

It has been shown (Nyburg & Potworowski, 1973) that knowing the structure of any one 'key' n-alkane (in this case n=18: Nyburg & Lüth, 1972), the unit-cell dimensions and crystal structures of all other members of an isostructural series can be predicted. The unit cells predicted in the original paper are related to those used by N & M by a, b, c → a, −b, −a + c. Only the latter type of cell is referred to here.

N & M give the d spacings and their indexing of the powder lines but not, unfortunately, the relative intensities.

This makes it difficult to know, in one important respect, to what extent our predictions are correct.

In the original paper (Nyburg & Potworowski, 1973) it was assumed that the triclinic parameters a, b and γ remained the same throughout the isostructural series. For the purpose of this note we have taken advantage of the known values of these parameters for n=6 (Norman & Mathisen, 1961), 8 (Mathisen, Norman & Pedersen, 1967), and 18, and used a smoothed interpolation for n values in between. The parameters obtained fit those assigned by N & M quite well except a and b for n=10 which have been changed accordingly. Of the remaining triclinic parameters, c and β were taken to be exactly as predicted in the earlier paper. The values of γ given by N & M do not vary smoothly

<table>
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<tr>
<th>n</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>α</th>
<th>β</th>
<th>γ</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>α</th>
<th>β</th>
<th>γ</th>
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<tr>
<td>10</td>
<td>4.20</td>
<td>4.75</td>
<td>13.57</td>
<td>93.4</td>
<td>81.8</td>
<td>105.6</td>
<td>4.25</td>
<td>4.81</td>
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<td>93.8</td>
<td>81.7</td>
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<td>4.81</td>
<td>16.12</td>
<td>93.3</td>
<td>79.9</td>
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<tr>
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<td>4.29</td>
<td>4.82</td>
<td>18.58</td>
<td>93.1</td>
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<td>107.0</td>
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<td>92.2</td>
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Table 2. Summary of discrepancies between N & M spacings and indices, and those predicted

<table>
<thead>
<tr>
<th>n</th>
<th>Spacings (Å) of lines</th>
<th>Lines (and intensities)</th>
<th>Differences in ascribed indices</th>
</tr>
</thead>
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<td>recorded by N &amp; M with their indices, but not predicted</td>
<td>predicted but not observed</td>
<td>(Δ in Fig. 1)</td>
</tr>
<tr>
<td>----</td>
<td>----------------------------------------------------------</td>
<td>--------------------------</td>
<td>-----------------</td>
</tr>
<tr>
<td>10</td>
<td>3.833, 012</td>
<td>013 (vw), 105 (vw)</td>
<td>N &amp; M (▼ in Fig. 1)</td>
</tr>
<tr>
<td>12</td>
<td>3.085, 112</td>
<td>101, (w), 106 (vw), 110 (ms)</td>
<td>N &amp; M</td>
</tr>
<tr>
<td>14</td>
<td>none</td>
<td>(012+101) (w), 013 (vw)</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>3.577, 102</td>
<td>001 (s)*, 003 (w), 004 (vw)</td>
<td></td>
</tr>
</tbody>
</table>

* Possibly obscured by beam-stop.
with \( n \) so we have taken our predicted values slightly modified to fit exactly the known values for \( n = 6, 8 \) and 18. The two sets of parameters are given in Table 1.

![Diagram](image-url)

Fig. 1. Indices, 2\( \theta \) values and relative intensities predicted and observed by N & M for powder diagrams of the \( n \)-alkanes \((n = 10, 12, 14, 16) \). See Table 2.

- Indices and measured 2\( \theta \) agreeing with those of N & M.
- Lines recorded by N & M but not predicted.
- Lines predicted but not observed by N & M.
- Observed and predicted positions agreeing but with indices different from those assigned by N & M.

It will be seen from Fig. 1 that the agreement between observed and predicted positions of the powder lines is on the whole very good for all four alkanes; where both the position and N & M’s indexing agree with our predictions we have indicated this by \( \alpha \). The discrepancies are of three kinds and are set out in Table 2. The most serious discrepancies concern the moderately strong reflexions (one each for \( n = 12, 14 \) and 16) which were not observed by N & M. We do not know the reason for this. It should be noted from Table 2 that for \( n = 16 \) there is virtually no agreement in the indexing. This is due to the differences in \( c \) and \( \beta \) parameters (Table 1). We agree with the \( y \) parameter assigned by N & M but are uncertain how this value was obtained by them since no \( hkl \) or \( h00 \) indices were assigned.

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References


**Notes and News**

*Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the Executive Secretary of the International Union of Crystallography (J. N. King, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England).*

**New volume of International Tables for X-ray Crystallography**

Volume IV, entitled *Revised and Supplementary Tables* and edited by James A. Ibers and the late Walter C. Hamilton, has just been published for the Union by The Kynoch Press, Witton, Birmingham B6 7BA, England, at a price of £10.00. Orders may be placed direct with The Kynoch Press or from Polycrystal Book Service, P.O. Box 11567, Pittsburgh, Pa. 15238, U.S.A., or with any bookseller.

Since the publication of Volume III in 1962, experimental and theoretical activity in all areas of crystallography has greatly increased. The principle motivation for a new volume was to provide revised values for atomic scattering factors, X-ray wavelengths and atomic absorption coefficients.

Volume IV has a cumulative index for all four volumes. It contains material in an earlier volume, the reference to the earlier volume is included in parenthesis. In such cases, the numerical values given in Volume IV should be used, but the earlier volume should also be consulted for the sometimes extensive textual material accompanying the tables.

A number of special topics, mainly mathematical in content, which were not included in Volume II, have developed considerably and have been incorporated in Volume IV. Such new material, selected by the Editors, includes diffractometer calculations, analysis of thermal motion in crystals, and some aspects of direct methods for phase determination. Although some of this material is more textual than tabular, it has been included because of its great importance to most structural crystallographers. Omission of other topics should not be taken as indicative of their relative unimportance. Selection had to be made by the Editors. The Union is greatly indebted to the Editors and to all the contributing authors for making the publication of this volume possible.

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