the approximation. It is this term $\Delta = C^2 - 4AB$ which determines both the quality of the shape of the peak and the accuracy with which the maximum position can be determined.

For $\Delta < 0$ the approximation is good and will yield accurate results provided $A < 0$, $B < 0$. If $\Delta < 0$, but $A > 0$ and $B > 0$ then the wrong point has been chosen as $Z(0, 0)$ and the peak obtained will be shifted from the true position by an amount proportional to the distortion in the elliptical paraboloid. This has been shown graphically by Dickens. As is correctly explained by Dickens, the maximum positions found using each of the points 616, 627, 634 in turn as $Z(0, 0)$ should not be expected to coincide. However, Dickens' use of these maximum positions as a measure of the accuracy of the L.K. approximation is invalid since the approximation only holds when the highest point, i.e. 645 is used as $Z(0, 0)$. Dickens' method of checking the accuracy of the L.K. method by choosing points other than the highest point on the map as $Z(0, 0)$ often will lead to substantially incorrect values. This can be shown by examining the
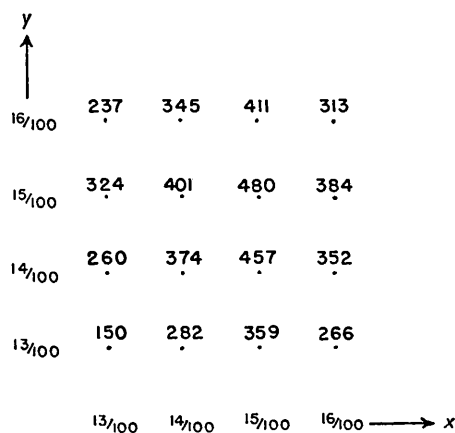

Fig. 1. Set of hypothetical electron-density values on (001)

following example (Fig. 1). In this case the highest mesh value is initially located at $x = y = 0\cdot1500$.

When the L.K. method is used with $Z(0, 0) = 480$, $\Delta = -16,093$ and the peak position obtained is $x_m = 0\cdot1495$, $y_m = 0\cdot1475$, which checks quite closely with graphical results. If the method is applied using 401 as $Z(0, 0)$ (in order to check the accuracy as Dickens has done), $\Delta = +199$ and the calculation reduces to an absurdity, i.e. the peak position obtained is $x_m = -0\cdot1834$, $y = 0\cdot1711$, a shift of almost thirty three electron density intervals to the left in x, and two electron density intervals upwards in y. This effect is due to the marked deviation of the peak shape from a true elliptical paraboloid. It is this latter result which is also to be expected when the L.K. method is used on unresolved atoms. The shapes of these unresolved peaks deviate considerably from elliptic paraboloidal character in the neighborhood of the peak maximas; in fact the saddle point type distribution is most likely to be present. Thus it is to be expected that 'enormous' errors will be involved (see above example); indeed the L.K. approximation was never intended for use on unresolved atoms.

There are two simple criteria which must be obeyed if the L.K. approximation is to be used correctly: (1) Both A and B must be less than 0. This can be ensured by using the highest point on the map for $Z(0, 0)$; (2) $\Delta < 0$, i.e. the values of $x_{\mathrm{max.}}$ and $y_{\mathrm{max.}}$ obtained using the L.K. method must each be less than one.

Lastly, the claim that obliquity of the mesh could be neglected if only peak heights are required, was based on calculations where θ was close to 90°. It is to be expected that if θ deviates considerably from 90°, the more detailed calculations must be used in order to obtain accurate results.

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The space groups of three isomers of acorone.

By E. J. Gabe, *Viriamu Jones Laboratory, University College, Cardiff, Wales, England*

Acorone is a sesquiterpene ketone whose empirical formula is $C_{15}H_{24}O_2$. It occurs in three stereoisomeric forms: acorone, isoacorone and neoacorone (Sorm, Herout & Sykora, private communication), whose structures are of considerable stereochemical interest. The data presented below are the results of a preliminary X-ray investigation of these isomers.

The cell dimensions and space groups summarized in Table 1 were determined by oscillation and Weissenberg photographs taken with Cu $K\alpha$ radiation. The accuracy of the values quoted is of the order of 1%.

Acorone

Acorone itself forms poor crystals with ill-defined faces; the Laue symmetry is $2/m$. The only systematic absences are $0k0$ for k odd and, as the substance is optically active, the space group is uniquely determined as $P2_1$. Compar-

Tabel 1. *Crystallographic data*

Substance	a (Å)	b (Å)	c (Å)	β	Density g.cm.$^{-3}$ Obs.	Calc.	Z	Space group
Acorone	6·2	14·2	8·3	96°	1·09	1·08	2	$P2_1$
Isoacorone	6·2	9·6	23·7	—	1·12	1·11	4	$P2_12_12_1$
Neoacorone	6·4	13·7	32·4	—	1·12	1·11	8	$P2_12_12_1$

ison of the observed and calculated densities shows that there is one molecule in the asymmetric unit.

Isoacorone

The crystals are long orthorhombic needles with well developed {011} and {010} faces. The systematic absences are $00l$, l odd, $0k0$, k odd and $h00$, h odd. These determine the space group uniquely as $P2_12_12_1$. Comparison of observed and calculated densities again shows one molecule in the asymmetric unit.

Neoacorone

The crystals are also long orthorhombic needles with the {011} and {010} faces well developed. The systematic absences are the same as for isoacorone, again determining the space group uniquely as $P2_12_12_1$; it was, however,

found that the substances had two molecules in the asymmetric unit, by comparison of the observed and calculated densities.

The crystal structure of UN by neutron diffraction. By MELVIN H. MUELLER and HAROLD W. KNOTT, *Argonne National Laboratory, Lemont, Illinois, U.S.A.*

UN and UC have been reported (Rundle, Baenziger, Wilson & McDonald, 1948) to be cubic with a NaCl rather than a ZnS type crystal structure. Although the contribution of the nitrogen or carbon to the diffracted X-ray intensity is small, sufficient differences of intensity were observed on powder patterns within several pairs of lines to support the NaCl type structure for UN and UC (Rundle *et al.*, 1948). Recently, additional X-ray and neutron diffraction data were obtained definitely confirming the NaCl type structure for UN.

The sample used for this study was obtained by two different methods. Part of the sample was made by the decomposition of UH_3 at 400 °C. with further heating at 1000 °C. under 1 atm. of purified nitrogen. Another part was made by heating massive uranium metal in a nitrogen atmosphere at 800 °C. followed by additional heating at 1200 °C.

The X-ray diffraction intensity data were obtained with an X-ray diffractometer using a finely powdered sample with a spinner. These intensities were found to be in better agreement for the NaCl rather than the ZnS type structure as evidenced by an R factor of 0·085 and 0·141, respectively, for these two structures. The R factor was determined according to the following relation, $R = \Sigma|I_o - I_c|/\Sigma I_o$. A lattice parameter of 4·890± 0·001 Å was obtained for this same material from a Debye–Scherrer powder pattern using a modified Cohen least-squares method.

In order to confirm the structure unequivocally a neutron diffraction pattern was obtained with the diffractometer described previously (Sidhu, Heaton & Zauberis, 1956). A relatively small sample of UN was used which filled a vanadium tube $\frac{1}{8}''$ in diameter $\times 1\frac{1}{2}''$ in length. A summary of the relative neutron intensities together with calculated values for the NaCl and ZnS type structures is given in Table 1. The value of $2B$ in the Debye–Waller temperature factor was determined from the slope of the plot of logarithm of I_o/I_c versus

Table 1. *Observed and calculated relative neutron diffraction intensities for* UN *with* $\lambda = 1·088$ Å

| | Observed | Calculated* | |
hkl	I/I_0	NaCl type	ZnS type
111	0·0	0·4	8·9
200	101·4	102·5	0·3
220	100·0	100·0	100·0
311	0·0	0·4	77·1
222	44·7	46·3	0·1
400	25·5	25·2	25·2
331	0·0	0·2	4·2
420	79·4	79·3	0·2
422	66·7	66·1	66·1
333, 511	0·0	0·2	12·9
440	20·4	24·3	24·3
531	0·0	0·2	45·7
442, 600	56·6	53·4	0·1
620	39·3	39·0	39·0

* $I \propto |F|^2 p(1/(\sin^2 \theta \cos \theta))\exp(-2B \sin^2 \theta/\lambda^2)$.

$(\sin^2 \theta)/\lambda^2$, using only those reflections whose calculated intensity was the same for both structures. No correction was made for absorption since the μr value for the UN sample used was very small; namely, 0·035. There is considerable difference in the calculated relative intensities for many of the reflections for the two types of structure, and it is therefore apparent that the best agreement is with the NaCl type. The R factor for this type structure is 0·024.

The B value of 0·51 Å^2 as obtained above was also used to calculate $\bar{\mu}$, the root-mean-square displacement of an average lattice point in a direction perpendicular to the reflecting plane, and Θ_M the characteristic temperature, according to the following given relation (James, 1948):

$$B = M/(\sin \theta/\lambda)^2 = 8\pi^2\overline{\mu^2} = \{6h^2T/(mk\Theta_M^2)\} \times \{\Phi(x) + x/4\} .$$