Table 4. U–O *bond lengths as function of bond strengths*

Uranyl bond		Six secondary bonds		Four secondary bonds	
s	Bond length	*s*	Bond length	*s*	Bond length
2·00	1·70 Å	0·33	2·49 Å	0·50	2·38 Å
1·75	1·74	0·42	2·43	0·63	2·30
1·50	1·83	0·50	2·38	0·75	2·23
1·25	1·95	0·58	2·33	0·88	2·15
1·00	2·08	0·67	2·28	—	—

is nearly normal (86°) to the associated uranyl axis. The distances and bond angles within the group agree well with those observed in other acetates.

The crude experimental determination of the hydrogen coordinates places the H_{III} atom in the same plane as the carbon and oxygen atoms, and this feature was retained in modifying the experimental coordinates for the hydrogen atoms so as to give more reasonable C–H distances and bond angles. H–O distances outside a given acetate group are all greater than 2·7 Å, showing that there is no real bonding.

The O_{III}–O_{IV} distances are 2·21±0·05 Å within and 2·76±0·05 Å between acetate groups. These are alternating edges of the nearly plane hexagon of oxygen atoms about the uranium atom shown in Fig. 2.

The Fourier syntheses were carried out on X-RAC, while all other calculations were made by hand. The authors are deeply grateful to Prof. Raymond Pepinsky for the generous and hospitable manner in which he made X-RAC available to us, and to the X-RAC personnel for their valuable help.

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An Account of some Computing Experiences

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Experience is summarized in the use of fractional shifts of scale, temperature and distance parameters, values of $r = \Sigma w(|F_o|^2-|F_c|^2)^2/\Sigma w|F_o|^4$, the behavior of temperature factors and a three-dimensional Patterson superposition program in the determination of a number of structures.

The increasing availability of high-speed digital computers in crystallography makes it desirable that general accounts of procedures and experience be available. Certain changes and developments in known methods, especially greater emphasis on three-dimensional methods, as well as the development of new methods, are taking place in a number of different laboratories. The variety of computers now available renders an account of programs for some particular computer of only special interest, but we feel that a more general statement of computer experience and techniques is of interest to crystallographers.

The Remington Rand 1103 UNIVAC SCIENTIFIC high-speed digital computer, in use for crystallographic computations in this laboratory for over three years, has a larger memory than computers for which similar computations have been described (Bennet & Kendrew, 1952; Ahmed & Cruickshank, 1953; Mayer & Trueblood, 1953; Thompson, Caminer, Fantl, Wright & King, 1954; Cochran & Douglas, 1955; Friedlander, Love & Sayre, 1955; Fowweather, 1955; Sparks, Prosen, Kruse & Trueblood, 1956) and is comparable in this respect with the IBM 704 for which some programs have been written (Vand, Turley & Pepinsky, 1957). We describe here some possibilities* in techniques and methods made possible by this large memory, with emphasis on the solutions of types of problems, rather than on the computer language and details.

1. Remarks on least-squares procedures

Since the introduction of the least-squares method into crystallography (Hughes, 1941) and the statistical treatment of errors (Hughes & Lipscomb, 1946; Cruickshank, 1949) these procedures have been widely used. They are particularly suitable for use in high-speed computers on which a large number of cycles of refinement can readily be made with relatively little output. Most of our comments arise from experience in which five to twenty cycles of refinement have been carried out on each of about fifteen different structure determinations during the past three years.

Our programs have been devised to minimize the function $E = \Sigma w(|F_o|^2-|F_c|^2)^2$. This function has the advantage over the more usual $D = \Sigma w(|F_o|-|F_c|)^2$ of continuous derivatives at $F_c = 0$, which makes it unnecessary to introduce arbitrary criteria for discarding reflections of uncertain phase. Also, the square-root operation for deriving $|F_c|$ for noncentric structures is avoided. Weighting factors $w = (4\,F_{\text{min.}}/F_o)^4$, when $4\,F_{\text{min.}} \leq F_o$, and $w = 1$ when $4\,F_{\text{min.}} \geq F_o$ (Lavine & Lipscomb, 1954) were used in most of our studies except as described below.

The aim of the least-squares refinement is to minimize E with respect to the scale factor k and the parameters that define the crystal structure. These parameters are the positional coordinates of all the atoms in the asymmetric unit and their thermal parameters. For the latter, two alternatives are normally available. An atom may be treated as isotropic, with temperature factor $\exp\,(-B\sin^2\theta/\lambda^2)$, in which B is a parameter; or it may be refined anisotropically, with temperature factor

$$\exp\,-(h^2\beta_1+k^2\beta_2+l^2\beta_3+hk\beta_4+kl\beta_5+lh\beta_6)\,,$$

all six β's being adjustable. In our general least-squares program, the choice between these alternatives can be made separately for each atom in the asymmetric unit, and this choice can be altered at will at the beginning of each new refinement cycle.

(A) *Fractional shifts*

Nearly all of our experience with the least-squares method has neglected the off-diagonal terms, which represent interactions between parameters. Only in one comparatively simple example, B_6H_{10}, have we studied their effect on total computing time. In this trial it appeared that the improvement in rate of convergence did not repay the added computing time per refinement cycle. It is probable that, provided the chosen crystal axes are orthogonal or nearly so and two-dimensional refinements are restricted to well-resolved projections, the off-diagonal terms can be safely neglected. Other groups have investigated the inclusion of part or all off-diagonal terms more extensively (Sparks & Trueblood, 1958). We feel that for a simple structure the total computing time can be reduced by the inclusion of off-diagonal terms but the total time is generally so small that it usually does not matter greatly. For a complex structure we feel that refinement will normally proceed more rapidly with the use of diagonal terms only. The demarkation between simple and complex structures depends upon the computer memory and seems to be of the order of 27 position parameters in our computer. When diagonal terms only are used it is necessary to introduce damping factors (n_k, n_b, n_x, respectively) to the changes in the scale, temperature, and distance parameters, at the end of each cycle of refinement (Dickerson, Wheatley, Howell & Lipscomb, 1957; Cromer, 1957). When an essentially correct trial structure is being refined, it is often wise to choose $n_b < n_x$ until the position parameters are well refined and then to increase both n_b and n_x. Typical values are $n_b = \frac{1}{8}$ and $n_x = \frac{1}{4}$ for the early stages of refinement and $n_b = \frac{1}{2}$, $n_x = \frac{1}{2}$ for the final stages of refinement. We presently choose $n_k = n_x$, usually, to increase the rate of refinement of the scale factor. Usually a larger n_b is permissible when anisotropic temperature factors are introduced. At the end of each cycle the parameter shifts should be carefully compared with shifts from the previous cycles. The value of n should be decreased if a number of shifts of appreciable magnitude oscillate in sign, and n should be increased if there are very few sign changes and convergence seems to be slow. Provision for separate n's for atoms of high atomic number or atoms with particularly anisotropic thermal motions is undoubtedly desirable. Often one may observe a few parameter shifts which are far greater than the average shifts and are not oscillating. If so, refinement has often been considerably hastened by the introduction of a few of the largest full shifts manually, provided that these large shifts do not produce oscillations of the signs of these shifts. The reverse procedure, setting $n_b > n_x$, is also useful, particularly at the trial structure stage. This procedure focuses attention on the thermal parameters, which often increase abnormally rapidly for incorrectly placed atoms in the trial structure, as described below.

(B) *Behavior of temperature factors*

The values of B in the isotropic temperature factor

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* A preliminary account of the use of the 1103 was made by Dickerson, Wheatley, Howell & Lipscomb (1957), and further developments were presented at the Fourth International Congress of Crystallography (Lipscomb, Dickerson & Hirshfeld, 1957).

Table 1. *Successive values of temperature parameters B and discrepancy factors R and r during the refinement of an incorrect model of* B_6H_{10}

The positional parameters, which were refined at the same time, are not shown. The last column gives final values for the correct model, in which a single H atom in a general position replaces the two atoms H_5 and H_7, which were placed on a mirror plane. Standard deviations of these B values are at least comparable with the absolute magnitudes of the negative values

Cycle	1	2	3	4	5	6	7	8	9	10	11	12	True structure
B_1	2·5	2·9	2·8	2·7	2·5	2·4	2·3	2·2	2·0	1·9	1·9	1·9	1·8
B_2	2·5	3·0	3·1	3·1	3·1	3·2	3·2	3·2	3·3	3·3	3·3	3·3	3·3
B_3	2·5	3·0	3·2	3·2	3·2	3·3	3·3	3·3	3·3	3·3	3·3	3·4	3·3
B_4	2·5	3·2	3·3	3·3	3·3	3·2	3·2	3·1	3·0	3·0	2·9	2·9	2·7
H_1	1·4	2·8	3·0	2·5	1·2	1·1	0·5	0·1	−0·8	−1·2	−1·3	−1·2	−0·9
H_2	1·4	0·7	0·2	−0·1	−0·3	−0·4	−0·5	−0·5	−0·4	−0·2	0·1	0·1	0·5
H_3	1·4	2·0	1·5	1·2	1·0	0·9	0·9	1·0	1·3	1·7	1·8	1·9	2·2
H_4	1·4	2·6	2·8	2·8	2·7	2·5	2·3	2·1	1·7	1·1	1·2	1·2	−0·5
H_6	1·4	0·8	0·2	−0·2	−0·5	−0·8	−1·0	−1·2	−1·5	−1·6	−1·5	−1·6	−1·2
H_5	1·4	5·5	9·8	14·7	19·6	25·5	30·0	32·9	37·8	40·7	29·4	35·4	5·3
H_7	1·4	4·0	6·0	8·1	10·3	13·4	17·0	21·5	37·4	69·9	73·9	97·5	
$100R$	19·0	13·2	13·2	12·9	12·7	12·4	12·2	12·0	11·6	11·3	11·4	11·3	9·9
$100r$	24·9	9·0	7·7	7·1	6·7	6·4	6·1	5·8	5·4	5·0	5·0	4·9	2·7

Damping factor $n_k = n_x = n_b$ 0·31 0·31 0·31 0·31 0·39 0·39 0·39 1·00 1·00 1·00 0·50

$\exp(-B \sin^2 \theta/\lambda^2)$ show very striking behavior upon repeated application of least-squares refinement. If, in an otherwise correct structure, some of the light atoms are incorrectly placed (perhaps inadvertently!) the values of B for the incorrectly placed atoms rise to very large values. Thus the procedure effectively eliminates these atoms from the trial structure. This procedure is particularly useful if the data are sufficiently incomplete that the corresponding electron density maps or difference maps have false peaks arising from convergence difficulties. The results for B_6H_{10}, in which H_5 and H_7 had been incorrectly placed, are very striking (Table 1). The values of B after a number of cycles of refinement of the correct structure are at least somewhat reasonable, as indicated in the last column of Table 1.

Another equally useful but less striking use of the temperature factor is the identification of C, N and O atoms in a trial structure where the chemical ambiguities do not allow a distinction. In ψ-conhydrine HBr, one of the atoms of a six-membered ring is N, while all others are C. After several cycles of three-dimensional least-squares refinement, in which all six atoms were assigned the same atomic scattering factor, the values of B became 4·1–4·7 for the five C atoms, but only 2·1 for the N atom. In this example, the refinement thus tended to place more electrons in the region of the peak of the N atom.

The behavior of temperature factors also suggests which atoms require large coordinate shifts. If an atom is, say, about 0·5 Å away from its true position, its B value will become anomalously large in the region of 5–10 Å^2, and the error in B decreases approximately proportionally to the error in the atomic position as the refinement proceeds. Refinement can then be accelerated if special attention is given to large shifts for these particular atoms in the early stages.

For the sake of completeness we may suggest that values of B will be quite useful in identification of atoms distributed at random over a number of sites greater than the number of atoms. However, our present experience does not include examples of this kind.

The behavior of temperature factors could conceivably make the least-squares procedure quite useful at earlier trial structure stages but a more extended study appears to be required, especially in the choice of suitable weights, number of reflections used, and perhaps the inclusion of cross-terms. In one extensive series of trials in the B_8Cl_8 structure determination, the behavior of B values was not as helpful as we had hoped: we found a fair number of near-homometric structures which failed to give clear indications of the incorrectly placed Cl atoms. Until further trials are made on as-yet-unsolved structures, one must have some reservations concerning the power of the method in this limited form at the early trial structure stage.

Of even greater value would be a procedure, now being studied here, of introducing a few small, broad electron-density maxima with scattering amplitudes,

$$\alpha f \exp\{2\pi i(hx+ky+lz)\} \times \exp -B\ (\sin^2 \theta/\lambda^2)$$

where α and B are parameters in the least-squares procedure, into a region where an atom is expected. We hope to find conditions under which the correct atoms will then refine toward reasonable values of α and B while the other, incorrectly placed, trial atoms will essentially vanish from the structure after a few cycles. This method should also prove quite valuable in the identification of atoms that have slightly differing atomic numbers, as in the ψ-conhydrine HBr case mentioned earlier.

(C) *Convergence*

Convergence is followed by noting the values and signs of the shifts, the value of $E = \Sigma w(|F_o|^2 - |F_c^2|)^2$ which is being minimized, the value of $r = E/\Sigma w|F_o|^4$,

Table 2. *Values of R and r for various structures*

Structure	Space group	R	r	Number of parameters	Number of reflections
Iresin diester (1)	$P2_1$	0·16	0·16	159	1430
Roussin black salt (2)	$P\bar{1}$	0·14	0·08	89	1395
BCl_3 (3)	$C6_3/m$	0·11	0·06	5	109
B_2F_4 (4)	$P2_1/n$	0·11	0·06	12	131
B_6H_{10} (5)	$Cmc2_1$	0·10	0·03	52	234
B_9H_{15} (6)	$P2_1/n$	0·16	0·13	37	374
B_4H_{10} (7)	$P2_1/n$	0·09	0·04	45	616
B_5H_{11} (7)	$P2_1/n$	0·11	0·05	51	298
B_8Cl_8 (8)	$P2_12_12_1$	0·17	0·14	105	1175
ψ-Conhydrine HBr (9)	$P2_12_12_1$	0·11	0·06	160	920
Ag *cyclo*octatetraene NO_3 (10)	$P2_1/a$	0·11	0·06	58	1136

(1) Rossmann & Lipscomb, 1959.
(2) Johansson & Lipscomb, 1957.
(3) Spencer & Lipscomb, 1958.
(4) Trefonas & Lipscomb, 1958.
(5) Hirshfeld, Eriks, Dickerson, Lippert & Lipscomb, 1958.
(6) Dickerson, Wheatley, Howell & Lipscomb, 1957.
(7) Moore, Dickerson & Lipscomb, 1957.
(8) Jacobson & Lipscomb, 1959.
(9) Yanai & Lipscomb, 1959.
(10) Mathews & Lipscomb, 1958.

and values of $R = \Sigma||F_o|-|F_c||/\Sigma|F_c|$ for all reflections and the classes $h0l$, $hk0$, $0kl$, h odd, k odd, l odd, $h+k$ odd, $k+l$ odd, $h+l$ odd and $h+k+l$ odd. These latter classes aid in the discovery of certain nearly-homometric structures. It is most important not to stop refinement when the parameter shifts become comparable with their standard deviations because a number of further cycles of refinement usually produces a cumulative shift many times these standard deviations. Although individual cases vary appreciably, the following table of values of R and r may be of some use for comparison when structures of comparable complexity are being refined (Table 2). In most of these examples, reduction of the sum of squares of residuals had been carried well beyond the limits imposed by such errors as precision of intensity measurements, accuracy of film factors, errors of correlation, neglect of extinction corrections (especially in the B_8Cl_8 work), absorption errors, etc.

Variation of weighting factor schemes in order to improve the rate of convergence (Vand & Pepinsky, 1957) or in order to minimize the final standard deviations (see below) are important areas in which our experience is so limited that we mention these points here only for completeness.

(D) *Errors*

The inclusion of weighting factors in the expressions for the standard deviations of bond distances is of great importance (Hughes & Lipscomb, 1946). Unfortunately, the Fourier method of computing standard deviations (Hughes & Lipscomb, 1946; Cruickshank, 1949) is not usually modified to include weights related in at least a general way to standard deviations of the observations. We have found that a least-squares refinement of $AgNO_3$ *cyclo*octatetraene with weights, as described by Lavine & Lipscomb (1954), gave standard deviations less by a factor of two than the standard deviations obtained from a similar refinement of the same compound but with the use of unit weights at every stage of the calculation, including the probable error computation. If the errors are truly random, it is a well-known result of the least-squares procedure that the 'best' system of weighting gives the lowest standard deviations. This result suggests a study, now under way, in which systematic weighting schemes are tested by variation of the weighting curve with such parameters as size of reflection, angle of scattering and number of times of observation. A limited study of some of these effects was made in the B_6H_{10} paper (Hirshfeld, Eriks, Dickerson, Lippert & Lipscomb, 1958).

(E) *Space group problems*

Our least-squares program is applicable to all triclinic, monoclinic and orthorhombic space groups. It is not necessary to write special least-squares programs for space groups of tetragonal, hexagonal and cubic symmetry. If a structure is refined in one of the subgroups of triclinic, monoclinic or orthorhombic symmetry, the final structure will, in fact, have the higher symmetry well within the standard deviations (Spencer & Lipscomb, 1958). Of course, the list of observed reflections must be expanded to the lower symmetry, and it is the exactness of equality of related reflections in this expanded list that causes the structure to deviate so little from its true symmetry when it is being refined in a subgroup.

2. Remarks on Fourier methods

Problems relating to weighting factors, convergence and computer output are more easily handled by least-squares procedures than by Fourier series methods on digital computers, and hence our use of three-dimensional Fourier series is not as extensive as is often the case.

In the trial structure stage, an important program is the point-by-point three-dimensional Patterson superposition program which selects the Buerger minimum function at each point (Buerger, 1951). In our experience, this is the most powerful and graphic

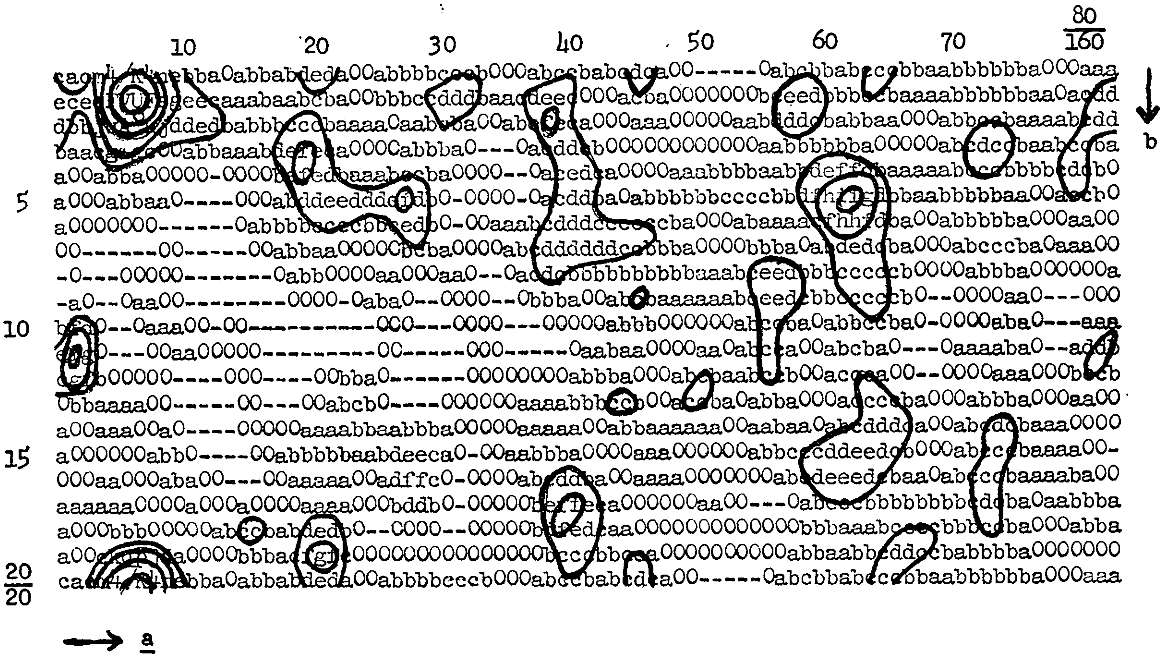

Fig. 1. Section at $\frac{18}{20}c$ of a three-dimensional Fourier summation for dibromobenzoateiresin. Phases are determined by bromines only. Contours are plotted at intervals of three letters, corresponding to a difference of 1 e.Å^{-3}, except in the vicinity of the bromine atom, where contours are plotted at intervals of 5 e.Å^{-3}. Special symbols are $l = +$ and $L = /$, in order to reduce confusion with 1.

method of obtaining trial structures. For a typical structure this program requires 64,000 words (one word is a 36-place binary number) of magnetic tape storage available in our model of the 1103 Computer. Any peak may be chosen for the superposition and any number of superpositions may be done. Thus a single interaction does not need to be found for the first superposition. This point-by-point program is much more sensitive and informative than its graphical equivalent. Moreover, it is much easier to depend upon small details in this map than in the more usual graphical maps obtained by superposition procedures. The great saving of time allows the trial of many different Patterson peaks as starting points for the superposition procedure.

Since output problems are of major importance in our computer and in many other computers, we feel some discussion of output is of merit, even though such a discussion would not be of interest to all readers.

Since the 1103 Computer loses no time when a single character is punched, provided the interval between punch commands is greater than 17 milliseconds, we use a single character to represent the electron density at each point of crystal space in the Fourier program (cf. Bennett & Kendrew, 1952). The use of small and large letters, numbers, and two colors in the typing, allows an interval of 128 in the sampling of the electron density before a repeat becomes necessary (Fig. 1). The color code can be chosen at any particular contour level so that it is not always necessary to draw contours on the maps printed by the typewriter. The color code can also be used to distinguish between positive and negative values of the electron density.

A three-dimensional difference map is computed at least once for each structure. Although full anisotropic temperature factors, with six constants per atom, are available in the least-squares program, we generally do not introduce this anisotropic refinement unless this difference map clearly indicates that these additional parameters are necessary. Frequently, therefore, these anisotropic temperature factors are introduced only for the heavy atoms. Similarly, hydrogen atoms are normally introduced only when some evidence for them is found in the difference map. Finally, it is quite usual that several three-dimensional Fourier series may be computed at the various trial structure stages.

3. Remarks on other procedures

There is little point in describing in detail a great number of auxiliary programs, which start with the observed data, make Lorentz-polarization corrections, correlate them, sharpen the data and compute the three-dimensional Patterson function. It has become customary to leave these data stored on magnetic tape and not to take them out of the computer at any of these intermediate stages. Even when trial structures are being tested and refined, the values of

E, r and R for various classes of reflections, and a running typewriter record of the number of reflections for which F_o differs from F_c by a factor of two, give some indication of correctness and of the stage of refinement, rather than the detailed lists of F_o and F_c. Detailed comparison of F_o and F_c is usually made only at the end of the investigation, with special attention to possible errors of F_o's which differ by more than a factor of two from the F_c's. Bond distance routines are also available, in which all interatomic distances less than some prescribed maximum, usually about 4 Å, are computed.

A conversion between anisotropic and isotropic temperature factors may, however, be of interest. This conversion makes use of the relations $\beta_1 = a^{*2}B/4$, $\beta_4 = a^*b^* \cos \gamma^* B/2$, and similar relations for β_2, β_3, β_5, and β_6. In order to fit an isotropic thermal parameter to an atom that was formerly refined anisotropically, the computer uses the approximation

$$\frac{B}{4}=\frac{a^{*2}\beta_1+b^{*2}\beta_2+c^{*2}\beta_3+2a^*b^*\cos\gamma^*\beta_4+2b^*c^*\cos\alpha^*\beta_5+2a^*c^*\cos\beta^*\beta_6}{a^{*4}+b^{*4}+c^{*4}+4a^{*2}b^{*2}\cos^2\gamma^*+4b^{*2}c^{*2}\cos^2\alpha^*+4a^{*2}c^{*2}\cos^2\beta^*}\,.$$

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