experimental thermal parameters and those calculated from a least-squares fit of individual experimental parameters to a rigid model. To perform this analysis we have carried out a Schomaker–Trueblood fit over individual experimental parameters and the resulting **T**, **L** and **S** are shown in Table 2. We have recalculated from them the individual parameters, whose comparison with the experimental ones gives us an idea of their deviation from rigid-body behaviour. The agreement factor found is $R = 0{\cdot}116$, only slightly lower than the lattice dynamical result, so the lattice dynamical calculations are encouraging in view of the simplified hypothesis that we have adopted in our dynamical model.

In Table 3 experimental thermal parameters can be seen, together with lattice dynamical and Schomaker–Trueblood fit results.

The present work is part of a research programme supported by the Government through the Comisión Asesora de Investigación Científica y Técnica.

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Acta Cryst. (1984). A**40**, 701–703

Note on Strengthened Translation Functions: the Positioning of a Well Oriented Molecular Fragment Using *DIRDIF* Procedures

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(*Received* 10 *April* 1984; *accepted* 2 *July* 1984)

Abstract

Strenghthened translation functions have been defined [Doesburg & Beurskens (1983). *Acta Cryst.* A**39**, 368–376] as convolutions of two electron density functions: *i.e.* the electron density representing the known fragment and the electron density obtained by the application of the *DIRDIF* procedures [Beurskens *et al.* (1982). In *Conformation in Biology*, edited by R. Srinivasan & R. H. Sarma. New York: Adenine Press]. Similar translation functions are defined as convolutions of the *DIRDIF* Fourier map with itself. The new functions are less powerful. The combination of the two types of functions, however, results in a more reliable method for the positioning of a fragment, if the fragment constitutes at least 10% of the total scattering power of the primitive unit cell. Examples of applications to known structures are given.

Introduction

The position of a correctly oriented fragment, represented by the electron-density function ρ_p, can be

0108-7673/84/060701-03$01.50

Table 1. *Test results for various structures*

Code name	Reference	Formula	Space group	Z	p^2*	Resulting peak number for the correct vector **q** in $Q(\mathbf{q})$	Resulting peak number for the correct vector **q** in $Q'(\mathbf{q})$
MONOS	(*a*)	$C_{15}H_{16}N_2O_2S$	$P2_12_12_1$	4	0·099	1	1
DIAMBE	(*b*)	$C_{13}H_{12}N_2O$	$P3_1$	3	0·327	1	1
					0·190	1	1
PENTAN	(*c*)	$C_{14}H_{14}N_2O_2$	*Pbca*	8	0·123	1	1
					0·083	1	3
HEPTA	(*d*)	$C_{30}H_{18}$	$P2_1$	4	0·246	1	1
					0·180	1	1
BRUCIN	(*e*)	$C_{33}H_{37}N_3O_4$	$P2_1$	4	0·145	1	3
					0·086	1	2

References: (*a*) Noordik, Beurskens, Ottenheijm, Herscheid & Tijhuis (1978); (*b*) van der Velden & Noordik (1980); (*c*) Doesburg, Noordik & Beurskens (1982); (*d*) Beurskens, Beurskens & van den Hark (1976); (*e*) Gould *et al.* (in preparation).

* p^2 is the relative scattering power of the input search fragment (not including symmetry-related fragments).

Table 2. *An example of the combination of* $Q_s(\mathbf{q})$ *and* $Q'_s(\mathbf{q})$ *for BRUCIN (see Table* 1); *two-dimensional search* (x, z) *for a twofold screw axis along* **b**

Results for $Q_{2_1}(x,z)$ Peak no.	W*	x	z	Results for $Q'_{2_1}(x,z)$ Peak no.	W'*	x	z	Combined result Peak no.	W^+ †	x	z
1	36	0·258	0·443	1'	28	0·090	0·261	1, 3'	31	0·258	0·444
2	31	0·417	0·237	2'	26	0·430	0·300	3, 1'	23	0·090	0·263
3	21	0·091	0·265	3'	22	0·258	0·445				
4	20	0·245	0·033	4'	19	0·492	0·042				
5	20	0·281	0·013	5'	18	0·273	0·320				

* Weight $W = 100\ Q_s/Q_0$.

† Weighted average: $W^+ = (2W + W')/3$.

determined by translation functions. The translation functions $Q_s(\mathbf{q})$, defined by Doesburg & Beurskens (1983), are based on the measure of fit of a search model ρ_{ps} (symmetry related to ρ_p) in the so-called *DIRDIF–P*1–Fourier map, denoted ρ_r (p = partial structure, r = remainder). For each symmetry element s, a function $Q_s(\mathbf{q})$ can be calculated and from the resulting maximum (at position **q**) the position of the fragment relative to the corresponding symmetry element is deduced. The strength of the functions $Q_s(\mathbf{q})$ results from the power of the *DIRDIF* procedures (Beurskens *et al.*, 1982).

For relatively small fragments (scattering power less than 20% of the scattering power of the entire primitive cell) the functions Q_s show subsidiary maxima, sometimes of the same height as the correct peak. On the other hand, the *DIRDIF–P*1–Fourier map usually gives more structural details than the collection of symmetry-related fragments ρ_{ps}. Therefore, we investigated the use of autoconvolutions of ρ_r for the location of the symmetry elements. This means that we now use the 'difference structure' as the search group instead of a known fragment.

Definition of the translation function $Q'_s(\mathbf{q})$

When using a known fragment ρ_p as input, the *DIRDIF* procedure will refine and extend the phases *and* recalculate the magnitudes of the difference structure factors, which results in a high-quality electron-density map ρ_r. If ρ_r is completely correct, ρ_r constitutes all symmetry-related fragments ρ_{ps} *and* all other unknown fragments. According to the general translation function (Argos & Rossmann, 1980) we define

$$Q'_s(\mathbf{q}) = \int_{\substack{\text{unit}\\ \text{cell}}} \rho_{rs}(\mathbf{r}-\mathbf{q})\rho_r(\mathbf{r})\,d\mathbf{r},$$

where

$$\rho_{rs}(\mathbf{r}_s) = \rho_r(\mathbf{r}) = \frac{1}{V}\sum_h F_r(\mathbf{h})\exp(-2\pi i\mathbf{h}\,.\,\mathbf{r})$$

and where the coefficients $F_r(\mathbf{h})$ are the structure factors of the difference structure, calculated by the *DIRDIF* procedure. The function is, of course, calculated in reciprocal space:

$$Q'_s(\mathbf{q}) = \frac{1}{V}\sum_{\mathbf{h}} F^*_{rs}(\mathbf{h})F_r(\mathbf{h})\exp(2\pi i\mathbf{h}\,.\,\mathbf{q})$$

where F^*_{rs} is the adjoint complex of F_{rs}, which is symmetry related to F_r.

The perfect overlap of a correct rest structure with itself will give

$$Q'_0 = \frac{1}{V}\sum_{\mathbf{h}} |F_r(\mathbf{h})|^2.$$

Results and discussion

The performance of the new translation functions was tested on various known structures. Some examples are given in Table 1.

The results show that the new functions Q_s' are less powerful than Q_s. In all given examples the highest peak in Q_s gives the correct position of the fragment, whereas for some of the examples peak number 2 or 3 in Q_s' gives the correct position. The new functions Q_s', however, are very useful if Q_s leads to an ambiguous result. An example is given in Table 2, in which the second peak in Q_s is ruled out because this peak does not occur in Q_s'.

An observation, made by Doesburg & Beurskens (1983), is also valid for Q_s': one-dimensional searches (for mirror or glide planes) are slightly more reliable than two-dimensional searches (symmetry axis) and far more so than a search for a center of symmetry.

The new translation functions are incorporated in the *DIRDIF* program package (Beurskens *et al.*, 1984), with negligeable increase in core and CPU requirements.

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Orientation Matrix Refinement During Four-Circle Diffractometer Data Collection

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(*Received 9 May* 1984; *accepted* 14 *June* 1984)

Abstract

Crystal movement, detected during data collection, requires the orientation matrix to be modified, so that reflection positions can be correctly predicted. If the unit cell is assumed to remain unchanged, the necessary modification is a small rotation of the matrix, *viz* premultiplication by an orthogonal matrix. This rotation is easily calculated from the observed positions of two or more centred reflections, by the application of quaternion algebra.

Introduction

Crystal movement (slippage) during single-crystal intensity data collection is a recognized problem in structure determination. It is usually monitored by the periodic measurement of a number of standard reflections. Changes in the intensities of these may indicate crystal movement, various forms of instrument instability or radiation damage to the crystal. If profile analysis techniques are employed in the data collection, changes in profile shape or position of the reflection peaks may also suggest movement.

Most commercial diffractometer control programs include a routine for re-establishing the orientation matrix and then continuing with the data collection, possibly repeating the last batch of reflections. This routine may be entered when standard reflection measurements show a significant change in orientation or at regular intervals as a precaution. The normal method is that described by Vandlen & Tulinsky (1971). Reflections contained in a list are centred and the orientation matrix is refined from the positions found. This refinement is generally an unconstrained refinement of the nine elements of the matrix (Tichý, 1970; Shoemaker & Bassi, 1970), which effectively changes the unit-cell parameters too.

The major drawback of this method is the time consumed by the centring routine, which can be particularly slow on machines not equipped with special hardware such as half-shutters (beam splitters). The

0108-7673/84/060703-02$01.50