Translation Functions: The Minimization of Structure-Independent Spurious Maxima

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Abstract

A procedure is described whereby the magnitudes of the structure-independent spurious maxima produced by translation syntheses are minimized. The new method has the advantage that it is applicable to all space groups. Structure-dependent spurious maxima are not as markedly reduced by the new method, but may be confidently suppressed by means of previously described procedures. Translation syntheses for which both sources of spurious maxima have been treated generally possess fewer strong peaks and flatter backgrounds. Trial calculations for test fragments comprising about half of the contents of the asymmetric unit typically produce a single solution vector which may be enhanced to ten times the magnitude of the next largest maxima. The usefulness of these procedures appears to diminish rapidly once the size of the search fragment is reduced to less than a quarter of the contents of the asymmetric unit. The solution vector may not be the largest maxima produced by these smaller fragments, but the translation maps will possess far fewer sizable spurious maxima to be confused with the solution vector than maps which have not been treated to reduce these spurious maxima.

Introduction

A previous analysis (Langs, 1975) has characterized the spurious maxima produced by translation syntheses as being either structure dependent or structure independent in nature. Structure-dependent spurious maxima form a centrosymmetric array of Patterson-like-vector maxima about the true solution vector of a translation synthesis. Previous attempts to eliminate these intramolecular vectors from various translation syntheses (Rossmann, Blow, Harding & Coller, 1964; Tollin, 1966; Crowther & Blow, 1967; Karle, 1972) were shown to be not completely successful. Recently a method has been described (Langs, 1985) for reliably eliminating these structure-dependent spurious maxima from translation syntheses that utilize rotational- or reflection-type symmetry elements, but these procedures were shown to be ineffective towards reducing the magnitude of structure-independent spurious maxima that occur in the same synthesis. A structure-independent spurious maximum is not related to any other peak in the translation function by a displacement that can be algebraically expressed purely in terms of the coordinates of the model. Although the appearance of such peaks is caused by the structure, a structural relationship among these spurious maxima cannot be demonstrated. The present work describes new methods that are useful in minimizing the magnitudes of these structure-independent spurious maxima.

Background

Molecularly deconvoluted Patterson-function coefficients may be shown to be of the following general form (Langs, 1975, equation 24)

$$\left[|E_{\mathbf{h}}|^2 - \sum_j^n \sum_p^m |E_{\mathbf{h},jp}|^2 \right]$$

$$= 2 \sum_{\substack{j,k \ p,q \\ (j \neq k \text{ if } p=q)}}^{n \quad m} (-1)^{2\mathbf{h} \cdot (t_j - t_k)} |E_{\mathbf{h},jp} E_{\mathbf{h},kq}|$$

$$\times \cos \left[2\pi (\mathbf{h}_j \cdot \mathbf{r}_p - \mathbf{h}_k \cdot \mathbf{r}_q) + \varphi_{\mathbf{h},jp} - \varphi_{\mathbf{h},kq} \right]. \qquad (1)$$

The diffraction amplitudes may be expressed either as $|F_{\mathbf{h}}|$ or as $|E_{\mathbf{h}}|$ as above. The summations are performed over the n symmetry positions of the space group for each of the m different search fragments in the asymmetric unit. The phased molecular transform computed from atomic coordinates of the pth oriented search fragment is identified as $E_{\mathbf{h},jp} = |E_{\mathbf{h},jp}| \exp i\varphi_{\mathbf{h},jp}$, with care being taken that the $|E|$ values are normalized with regard to the entire contents of the primitive equivalent unit cell. The vector $\mathbf{h}_j$ is related to $\mathbf{h}$ by the transpose of the inverse of the rotation matrix, R_j, of the jth equivalent position. The translation symmetry operator for the jth equivalent position is given as t_j. The molecular displacement vectors of interest, $\mathbf{r}_p$, are related to the initial trial coordinates, $\mathbf{r}_{jp}$, and crystallographic coordinates, $\mathbf{r}_j$, as $\mathbf{r}_p = \mathbf{r}_j - \mathbf{r}_{jp}$. The determinable quantity given as the left-hand side of (1), defined as

$$G_{\mathbf{h}} = \left[|E_{\mathbf{h}}|^2 - \sum_j^n \sum_p^m |E_{\mathbf{h},jp}|^2 \right],$$

is a signed real value referred to as the translation-function coefficient. Subsequent translation syntheses

$$T(\mathbf{r}) = \sum_{\mathbf{h}} (-1)^{2\mathbf{h} \cdot (t_j - t_k)} G_{\mathbf{h}} \cos \left[2\pi \mathbf{h} \cdot \mathbf{r} + \varphi_{\mathbf{h},jp} - \varphi_{\mathbf{h},kq} \right],$$

$$(2)$$

which are expected to produce a solution vector at $r = R_j^{-1} . r_p - R_k^{-1} . r_{q'}$ are limited in their usefulness primarily by the degree with which G_h approximates any particular cosine term chosen from the right-hand side of (1) to indicate the desired synthesis, that is

$$G_h \simeq 2(-1)^{2h \cdot (t_j - t_k)} |E_{h_j p} E_{h_k q}|$$
$$\times \cos [2\pi(h_j . r_p - h_k . r_q) + \varphi_{h_j p} - \varphi_{h_k q}]. \quad (3)$$

This approximation becomes progressively worse as the number of primitive equivalent symmetry positions of the space group increases and the relative size of the known molecular fragment decreases. In such instances both the magnitude and sign of G_h are at best a poor approximation for the phased molecular transform product indicated.

A second source of the structure-independent random noise in translation syntheses has previously been indicated (Langs, 1975, equations 15-17) as a consequence of our inability to estimate the corresponding imaginary component of the molecular transform product, which is a priori not known, i.e.

$$S_h = 2(-1)^{2h \cdot (t_j - t_k)} |E_{h_j p} E_{h_k q}|$$
$$\times \sin[2\pi(h_j . r_p - h_k . r_q) + \varphi_{h_j p} - \varphi_{h_k q}]. \quad (4)$$

This second source of structure-independent random noise leads to spurious maxima being produced at positions defined by trigonometric terms of the form $\cos[2\pi(h_j . r_p + h_k . r_q) + 2(\varphi_{h_j p} - \varphi_{h_k q})]$. These spurious indications would not normally be expected to reinforce one another strongly owing to the random nature of the value of the phase of the molecular transform. However, one can completely eliminate such spurious maxima by computing a translation synthesis over both the real and imaginary components of the indicated molecular transform product

$$T'(r) = \sum_h (-1)^{2h \cdot (t_j - t_k)}$$
$$\times \{G_h \cos [2\pi h . r + \varphi_{h_j p} - \varphi_{h_k q}]$$
$$- S_h \sin [2\pi h . r + \varphi_{h_j p} - \varphi_{h_k q}]\}. \quad (5)$$

Unfortunately it appeared that such syntheses could not be utilized since only the magnitude of S_h could be estimated directly from G_h,

$$|S_h| \simeq [4|E_{h_j p} E_{h_k q}|^2 - G_h^2]^{1/2}, \quad (6)$$

on the condition that

$$|G_h| < 2|E_{h_j p} E_{h_k q}|, \quad (7)$$

and the sign of the imaginary component could not be ascertained without the knowledge of the displacement vectors, r_p, which were a priori unknown. The following analysis will detail procedures that will be effective in reducing the magnitudes of the structure-independent spurious maxima arising from the two sources described above.

Analysis

For the purposes of this study it was thought to be useful to derive a probability expression that could describe the accuracy with which G_h estimates the value of any particular molecular transform product selected from the identity (1) as indicated by (3). The mean and variance of a particular molecular transform product

$$x = 2|E_{h_j p} E_{h_k q}| \cos [2\pi(h_j . r_p - h_k . r_q) + \varphi_{h_j p} - \varphi_{h_k q}] \quad (8)$$

may be shown to be

$$\mu(x) = \langle x \rangle = (-1)^{2h \cdot (t_j - t_k)} G_h \quad (9)$$
$$\sigma^2(x) = \langle x^2 \rangle - \mu(x)^2$$
$$= 2\left\{ \sum_{j,k,p,q} |E_{h_j p'} E_{h_k q'}|^2 \right.$$
$$\left. + (M/N)\left[\sum_{j,p} |E_{h_j p'}|^2 + (M-1)/2N \right]\right\}, \quad (10)$$

where the right-hand side of (10) includes all transform products except the particular term that is evaluated. The variance allows for the contribution of M residual atoms, out of the total of N equivalent atoms in the primitive unit cell, which are unassociated with any correctly oriented molecular fragment (see Langs, 1975, equation 24). A probability density function can be formulated on the assumption that the distribution of errors is normal:

$$P(x) = \exp \{-[x - \mu(x)]^2/2\sigma^2(x)\},$$

and may be shown to be of the form

$$P(x) = K \exp [\alpha \cos \theta - \beta \cos 2\theta] \quad (11)$$

where

$$\alpha = 2|E_{h_j p} E_{h_k q}|(-1)^{2h \cdot (t_j - t_k)} G_h/\sigma^2(x)$$
$$\beta = |E_{h_j p} E_{h_k q}|^2/\sigma^2(x)$$
$$\theta = 2\pi(h_j . r_p - h_k . r_q) + \varphi_{h_j p} - \varphi_{h_k q}.$$

The probability that the sign of G_h is correct may then be expressed as

$$P(G_h) = \frac{1}{2} + \frac{1}{2} \int_0^{2\pi} P(x) \cos \theta \, d\theta / \int_0^{2\pi} P(x) \, d\theta$$
$$= \frac{1}{2} + \frac{1}{2} \sum_{n=0}^{\infty} (-1)^n I_{2n \pm 1}(|\alpha|)$$
$$\times I_n(\beta) \left/ \sum_{-\infty}^{\infty} (-1)^n I_{2n}(|\alpha|) I_n(\beta). \right. \quad (12)$$

This probability will tend toward certainty as the size of the search fragment increases for structures in space groups possessing two equivalent positions, as the variance (10) reduces to the last two terms, which are dependent on M/N and will equal zero when the fragment comprises the entire contents of the

Table 1. *Total number of G_h found and number of G_h assigned incorrect signs for three known fragments for tetrahymanol hemihydrate at different probability levels*

The number of G_h expected to be wrong is computed as $\sum [1 - P(G_h)]$ over the total number of G_h above the probability threshold. The last three columns refer to the probability of the sign of G_h computed from the first 14-atom fragment given the additional information of the orientation of a second 14-atom fragment that does not affect the phase of G_h but rather its variance given by (10).

$\langle P(G_h) \rangle$	31-atom fragment			14-atom fragment			14 atoms plus 14		
	Total	Wrong	Expected	Total	Wrong	Expected	Total	Wrong	Expected
0·90	24	5	1	6	1	0	11	1	0
0·80	121	24	16	38	9	5	38	6	4
0·70	230	70	70	85	24	17	93	21	18
0·60	1047	292	329	274	95	86	339	115	107
0·50	5020	2059	2177	5020	2383	2368	5020	2374	2351

asymmetric unit. The variance will not equal zero for the analogous situation in higher-order space groups of primitive order n, since there are $(n+1)(n-2)/2$ non-vanishing terms represented by the first summation in (10), and a small variance will depend on a large value for $|E_{h_jp}E_{h_kq}|$ and small values for all other products $|E_{h_{j'}p'}E_{h_{k'}q'}|$.

Phase relationships

A probability estimate for the correctness of the phase of G_h having been formulated, it appeared logical to investigate whether interdependent relationships among the various G_h might provide a basis for determining which among these values are incorrectly phased. A number of different phase relationships can be easily derived as exact trigonometric identities, of which the following relationship appears to be significantly better than others investigated:

$$G_h = A_h[(G_k G_l - S_k S_l) \cos \Phi_{h,k}$$
$$+ (G_k S_l + S_k G_l) \sin \Phi_{h,k}]/A_k A_l, \quad (13)$$

$$S_h = A_h[(G_k G_l - S_k S_l) \sin \Phi_{h,k}$$
$$- (G_k S_l + S_k G_l) \cos \Phi_{h,k}]/A_k A_l \quad (14)$$

and

$$\theta_{h_{jpkq}} = [2\pi(\mathbf{h}_j \cdot \mathbf{r}_p - \mathbf{h}_k \cdot \mathbf{r}_q) + \varphi_{h_jp} - \varphi_{h_kq}]$$
$$= \tan^{-1} (S_h/G_h), \quad (15)$$

where the G_h are initially assumed to equal the particular molecular transform product indicated by (3), the vector triple $\mathbf{h} + \mathbf{k} + \mathbf{l} = 0$, and A_h and $\Phi_{h,k}$ are $2|E_{h_jp}E_{h_kq}|$ and $\varphi_{h_jp} - \varphi_{h_kq} + \varphi_{k_jp} - \varphi_{k_kq} + \varphi_{l_jp} - \varphi_{l_kq}$ respectively. Given the initial sign indications of a set of G_h, selected such that their individual probabilities (12) exceed some threshold, say 0·75, one can immediately re-estimate values of G_h and obtain *ab initio* values for S_h from the dominant terms

$$G_h \simeq KA_h\langle G_k G_l \cos \Phi_{h,k} \rangle_k / \langle A_k A_l \rangle_k \quad (16)$$

and

$$S_h \simeq KA_h\langle G_k G_l \sin \Phi_{h,k} \rangle / \langle A_k A_l \rangle_k, \quad (17)$$

here averaged over a large number of triple relation-

ships to minimize the error in the approximation. Thus one can determine not only which of the G_h must have their signs reversed, but, in addition, the phase of the imaginary component of the molecular transform product, *i.e.* $\tan^{-1}(S_h/G_h)$, which will enable us to compute the improved translation synthesis indicated by (5). The initial estimates of G_h and S_h can be refined by (15), again by averaging over triple relationships, as indicated in (16) and (17). Initial test calculations based on this scheme are very encouraging.

Trial calculations

The initial trial calculations were performed on the structure of tetrahymanol hemihydrate (Langs, Duax, Carrell, Berman & Caspi, 1977), $C_{30}H_{52}O \cdot \frac{1}{2}H_2O$, monoclinic, $P2_1$, $a = 7·42$, $b = 11·43$, $c = 30·90$ Å, $\beta = 101·9°$, $Z = 4$; the data set comprised 5020 independent reflections. The test conditions included (1) the knowledge of the orientation of one of the two crystallographically independent tetrahymanol molecules (31 of the 63 non-hydrogen atoms), and (2) the A, B and C rings of that molecule (14/63 atoms). Table 1 presents the number of total and incorrectly signed G_h that are found above a given probability threshold computed by (12) for each of these two test cases, plus an additional example of the phases computed from the 14-atom fragment of one molecule given the additional information of the orientation of a similar 14-atom fragment of the second molecule. The translation coefficients to determine the position of the 2_1 screw axis were defined as

$$G_{hkl} = [|E_{hkl}|^2 - |E_{hklp}|^2 - |E_{h\bar{k}lp}|^2 - M/N],$$

where M represents the residual number of atoms not defined by the fragment among the $N = 63$ atoms in the asymmetric unit. The probabilities for the correctness of the signs of these G_h were computed and all those coefficients for which the probability exceeded a chosen threshold were used as input to (16) and (17). The subgroup of G_h for which (16) indicated a reversal of sign was ranked on the magnitude of the product of $A_h\langle A_k A_l \rangle_k |G_h\langle G_k G_l \rangle_k|$, and the signs of a chosen fraction of these G_h, usually two

Table 2. *Iterative phase refinement for two known fragments for tetrahymanol hemihydrate at selected probability levels*

The fourth column indicates the number of G_h above the indicated probability threshold followed by the actual number of incorrectly signed values enclosed in parentheses. Subsequent columns indicate the progress for each cycle of refinement: firstly the number of G_h for which a sign reversal is indicated and performed, followed in parentheses by the actual number of these terms that are incorrectly changed in sign, followed by the resultant number of incorrectly signed G_h as a consequence of the operation.

Trial	Atoms	$P(G_h)$	Number of G_h (Wrong)	Cycle 1	Cycle 2	Cycle 3	$\langle\cos \Delta\theta\rangle$
1	31/63	0·63	759 (195)	138 (3), 63	47 (4), 24	15 (5), 19	0·982
2	31/63	0·58	1499 (444)	311 (4), 141	106 (8), 51	35 (14), 44	0·990
3	31/63	0·55	2293 (775)	531 (6), 256	182 (18), 110	66 (22), 95	0·986
4	14/63	0·57	1392 (594)	124 (17), 504	137 (26), 419	86 (29), 391	0·373
5	14/63	0·55	1826 (788)	136 (12), 676	197 (35), 549	121 (33), 494	0·427

Table 3. *Summary of the actual and relative peak heights produced by the various translation syntheses described in the text*

The relative peak height of the correct solution maximum is taken to be 100.

Peak	Trial 3				Trial 5			
	Φ_1	Φ_1'	Φ_0	Φ_0'	Φ_1	Φ_1'	Φ_0	Φ_0'
1	55 (100)	175 (100)	160 (100)	337 (100)	22 (100)	85 (118)	51 (100)	138 (104)
2	17 (31)	44 (25)	41 (26)	33 (10)	21 (97)	80 (112)	36 (70)	133 (100)
3	16 (29)	40 (23)	36 (23)	25 (7)	20 (92)	72 (100)	35 (68)	121 (92)
4	15 (27)	31 (18)	36 (23)	21 (6)	17 (80)	49 (68)	32 (63)	45 (34)
5	15 (27)	26 (15)	33 (21)	21 (6)	16 (73)	37 (52)	31 (62)	44 (33)
10	12 (22)	20 (11)	30 (19)	13 (4)	12 (56)	25 (34)	29 (58)	25 (19)
50	7 (13)	6 (3·4)	21 (13)	6 (2)	8 (35)	10 (14)	22 (43)	13 (10)

thirds based on the largest values of this product, were actually reversed prior to the next cycle of refinement. In no instance were the original magnitudes of G_h altered, and the magnitude of S_h was held at its fixed value determined by (4). Examples of these refinements presented in Table 2 are detailed with respect to both the number of wrongly signed G_h that have been successfully corrected and the degree of refinement convergence as measured by the $\langle\cos(\theta_{cal} - \theta_{obs})\rangle$, where the true phase of the molecular transform product, θ_{obs}, equals $\theta_{h_{jkpq}}$, which is known from the solved structure.

Both the phase-modulated translation synthesis (Langs, 1975)

$$\Phi_1(x, z)$$

$$= \sum_h (-1)^k G_{hkl} \cos [4\pi(hx + lz) + \varphi_{hklp} - \varphi_{h\bar{k}lp}]$$

and the improved synthesis, which minimizes structure-dependent spurious maxima (Langs, 1985),

$$\Phi_0(x, z) = \sum_{h,l} \cos [4\pi(hx + lz) + \theta_{hl}],$$

where

$$\cos \theta_{hl} = A_{hl}/[A_{hl}^2 + B_{hl}^2]^{1/2},$$

$$\sin \theta_{hl} = B_{hl}/[A_{hl}^2 + B_{hl}^2]^{1/2},$$

$$A_{hl} = \sum (-1)^k G_{hkl} \cos (\varphi_{hklp} + \varphi_{h\bar{k}lp})$$

and

$$B_{hl} = \sum (-1)^k G_{hkl} \sin (\varphi_{hklp} + \varphi_{h\bar{k}lp})$$

were used to assess how well the phase refinements reduced the sources of structure-independent spurious maxima. Results of these translation syntheses are given in Table 3, where Φ_0' and Φ_1' denote the functions computed with the refined phases of the molecular-transform product.

Discussion

The probability estimates computed by (12) appear to match the observed error frequency for the sign of G_h as is shown in Table 1. Remembering that all $5020 G_h$ would be absolutely determined with the correct sign if the correctly oriented search fragment comprised the complete contents of the asymmetric unit, *i.e.* all 63 atoms, it is somewhat surprising that less than 1% of the $5020 G_h$ have probabilities exceeding 0·9 in the most favorable example in the table where 31 of the 63 atoms comprise the search fragment. This situation becomes far worse with smaller fragments, where with a quarter of the structure less than 1% of the data have a probability of being 80% correct. This situation is not appreciably improved even when additional information such as the orientation of a second 14-atom fragment is incorporated in the computation of the variance (10).

Given this disappointing early prognosis, the results of the phase refinements shown in Table 2 are most remarkable. The first three trials examine different probability cuts for the 31-atom search fragment in an attempt to access the top 750, 1500 and $2500 G_h$.

The first trial selected the top $759G_h$, of which 195, or 25%, were noted to be incorrectly signed from the known structure. The first cycle of phase refinement indicated *via* (16) that 203 of the 759 terms were incorrectly signed, and of these 203 indications, 183 were actually noted to be in error. Thus in one cycle one could reduce the number of sign errors from 195 in 759, or 25%, to 32 in 759, or 4%. Although the results of a single refinement cycle were as optimistic for trials 2 and 3, a more cautious rate of refinement was found to be required in trials 4 and 5 in which the fragment size was reduced to 14 atoms, or a quarter of the structure. In trial 4 the probability threshold was lowered to 0·55 to obtain $1392G_h$, of which 594 or 43% were incorrectly signed. The first cycle of phase refinement indicated that 586 terms were incorrectly signed, but of these only 390 were truly the wrong sign, and the 196 remaining indications were in error. Attempts to refine these phases by blindly applying the indicated sign shifts does not succeed as was the case in the first three trials, and a more conservative rate of refinement was required to achieve some degree of phase-refinement convergence toward improved values. If the initial 586 indications, however, were ranked on the magnitude of the product $A_h\langle A_k A_l\rangle_k |G_h\langle G_k G_l\rangle_k|$, as previously indicated, the error rate may be seen to be less for those G_h for which this product is large. Thus in the top 124 ranked terms, only 17, or 14%, were found to be in error, as is noted in Table 2, and applying these sign shifts reduces the number of incorrectly signed G_h from 594 to 504. Successive cycles of refinement may be seen to reduce the number of mis-signed G_h from 594 to 391 with a root-mean-square phase error of more than 68° indicated by the average cosine value of 0·373 given in the table. These results appear to be much worse than those indicated in the first three trials in which the number of incorrectly signed G_h has been easily reduced by a factor of ten, and the phase error reduced to less than 10° on average.

Although Table 2 indicates that the phasing procedures that have been utilized appear to have deteriorated rapidly upon reducing the fragment size from 50 to 25%, the inspection of translation syntheses suggests that this deterioration is probably no worse with regard to finding a translation solution than had the phases not been refined. Table 3 summarizes the actual and relative sizes of peaks, relative to the correct solution vector for trials 3 and 5 indicated in Table 2. Note that Φ_1 and Φ_1' for trial 3 indicate that the actual magnitude of the solution vector has increased from 55 to 175, while background levels measured at peaks 50 have remained about the same. This improvement in the solution-vector-to-background ratio is totally a consequence of better phasing as the amplitudes in both syntheses are exactly the same. The corresponding Φ_0 synthesis produces no significant improvement over the Φ_1 computation, indicating that the spurious maxima are strongly structure-independent in nature. A comparison between Φ_0 and Φ_0' clearly indicates that the phase refinement has reduced the magnitudes of the structure-independent spurious maxima such that the solution vector is ten times larger than the second largest peak in the map. The Φ_0 map had more than 50 peaks above this threshold. The situation for trial 5 is similar in that phase refinement has markedly reduced the number of spurious maxima that are within 35% of the magnitude of the true solution vector. There are 50 such peaks in the Φ_1 synthesis compared with only ten such peaks in the corresponding Φ_1' map, and only three such peaks in the Φ_0' map compared to more than 50 in the corresponding Φ_0 map. Although it should be fairly obvious that the Φ_1' and Φ_0' syntheses did not converge to an unambiguous solution as a consequence of the phase refinement, one should not conclude that the exercise was without merit since the Φ_1 and Φ_0 maps happen to yield the correct solution as the largest peak. These tests show that these procedures do indeed minimize the influence of structure-independent spurious maxima in translation syntheses. Furthermore, one can be hopeful that better procedures can be foreseen for appropriately weighting the triplet terms that contribute to the refinement of the phases indicated in (13)–(15), as do better criteria for ascertaining which of those refined phases are most reliably determined.

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