International Tables for Crystallography (2006). Vol. F, Table 16.1.8.1, p. 339.

abrupt increase in correct peaks occurs when Fourier refinement is started.

Since the correlation coefficient is a relatively absolute figure of merit (given atomic resolution, values greater than 65% almost invariably correspond to correct solutions), it is usually clear when *SHELXD* has solved a structure. The current version of *SHELXD* includes an option for calculating it using the full data every 10 or 20 internal loop cycles, and jumping to the external loop if the value is high enough. Recalculating it every cycle would be computationally less efficient overall.

16.1.8. Applying dual-space programs successfully

The solution of the (known) structure of triclinic lysozyme by *SHELXD* and shortly afterwards by *SnB* (Deacon *et al.*, 1998) finally broke the 1000-atom barrier for direct methods (there happen to be 1001 protein atoms in this structure!). Both programs have also solved a large number of previously unsolved structures that had defeated conventional direct methods; some examples are listed in Table 16.1.8.1. The overall quality of solutions is generally very good, especially if appropriate action is taken during the Fourier-

Table 16.1.8.1. *Some large structures solved by the Shake-and-Bake method*

Previously known test data sets are indicated by an asterisk (*). When two numbers are given in the resolution column, the second indicates the lowest resolution at which truncated data have yielded a solution. The program codes are *SnB* (S) and *SHELXD* (D).

(*a*) Full structures (>300 atoms).

Compound	Space group	N_u (molecule)	N_u + solvent	N_u (heavy)	Resolution (Å)	Program	Reference
Vancomycin	$P4_32_12$	202	258	8Cl	0.9–1.4	S	[1]
			312	6Cl	1.09	D	[2]
Actinomycin X2	$P1$	273	305	—	0.90	D	[3]
Actinomycin Z3	$P2_12_12_1$	186	307	2Cl	0.96	D	[4]
Actinomycin D	$P1$	270	314	—	0.94	D	[4]
Gramicidin A*	$P2_12_12_1$	272	317	—	0.86–1.1	S, D	[5]
DMSO d6 peptide	$P1$	320	326	—	1.20	S	[6]
Er-1 pheromone	$C2$	303	328	7S	1.00	S	[7]
Ristocetin A	$P2_1$	294	420	—	1.03	D	[8]
Crambin*	$P2_1$	327	423	6S	0.83–1.2	S, D	[9], [10]
Hirustasin	$P4_32_12$	402	467	10S	1.2–1.55	D	[11]
Cyclodextrin derivative	$P2_1$	448	467	—	0.88	D	[12]
Alpha-1 peptide	$P1$	408	471	Cl	0.92	S	[13]
Rubredoxin*	$P2_1$	395	497	Fe, 6S	1.0–1.1	S, D	[14]
Vancomycin	$P1$	404	547	12Cl	0.97	S	[15]
BPTI*	$P2_12_12_1$	453	561	7S	1.08	D	[16]
Cyclodextrin derivative	$P2_1$	504	562	28S	1.00	D	[17]
Balhimycin*	$P2_1$	408	598	8Cl	0.96	D	[18]
Mg-complex*	$P1$	576	608	8Mg	0.87	D	[19]
Scorpion toxin II*	$P2_12_12_1$	508	624	8S	0.96–1.2	S	[20]
Amylose-CA26	$P1$	624	771	—	1.10	D	[21]
Mersacidin	$P3_2$	750	826	24S	1.04	D	[22]
Cv HiPIP H42Q*	$P2_12_12_1$	631	837	4Fe	0.93	D	[23]
HEW lysozyme*	$P1$	1001	1295	10S	0.85	S, D	[24], [25]
rc-WT Cv HiPIP	$P2_12_12_1$	1264	1599	8Fe	1.20	D	[23]
Cytochrome c3	$P3_1$	2024	2208	8Fe	1.20	D	[26]

(*b*) Se substructures (> 25 Se) solved using peak-wavelength anomalous-difference data.

Protein	Space group	Molecular weight (kDa)	Se located	Se total	Resolution (Å)	Program	Reference
SAM decarboxylase	$P2_1$	77	20	26	2.25	S	[27]
AIR synthetase	$P2_12_12_1$	147	28	28	3.0	S	[28]
FTHFS	$R32$	200	28	28	2.5	D	[29]
AdoHcy hydrolase	$C222$	95	30	30	2.8–5.0	S	[30]
Epimerase	$P2_1$	370	64	70	3.0	S	[31]

References: [1] Loll *et al.* (1997); [2] Schäfer *et al.* (1996); [3] Schäfer (1998); [4] Schäfer, Sheldrick, Bahner & Lackner (1998); [5] Langs (1988); [6] Drouin (1998); [7] Anderson *et al.* (1996); [8] Schäfer & Prange (1998); [9] Stec *et al.* (1995); [10] Weeks *et al.* (1995); [11] Usón *et al.* (1999); [12] Aree *et al.* (1999); [13] Prive *et al.* (1999); [14] Dauter *et al.* (1992); [15] Loll *et al.* (1998); [16] Schneider (1998); [17] Reibenspiess (1998); [18] Schäfer, Sheldrick, Schneider & Vértesy (1998); [19] Teichert (1998); [20] Smith *et al.* (1997); [21] Gessler *et al.* (1999); [22] Schneider *et al.* (2000); [23] Parisini *et al.* (1999); [24] Deacon *et al.* (1998); [25] Walsh *et al.* (1998); [26] Frazão *et al.* (1999); [27] Ekstrom *et al.* (1999); [28] Li *et al.* (1999); [29] Radfar *et al.* (2000); [30] Turner *et al.* (1998); [31] Deacon & Ealick (1999).

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