

For all 958 non-hydrogen protein atoms, including the calcium ion, the r.m.s. difference is 0.66 Å.*

Concluding remarks

The final proof in testing an X-ray detector is the collection and processing of crystal diffraction data, the determination of the structure and the refinement of that structure. The successful refinement of the BPLA₂ structure in methanol using data collected with a FAST diffractometer shows several things. Firstly, the TV area detector has now reached an operational state producing high-resolution data of good quality. The data were collected with the instrument in the diffractometer mode, at a speed of 1700 reflections h⁻¹ which is quite good. Furthermore, the very complex software (Thomas, 1982) works satisfactorily. Data reduction on the PDP 11/24 took 120 h. This is rather long. It is determined by the efficiency of the reduction program and the memory and speed of the computer. The latter has now been replaced by a VAX 11/730. The hardware has also been further improved. Subsequent to the work described in this article we have used the updated instrument in the film mode with the Munich area detector NE software package MADNES (Pflugrath & Messerschmidt, 1985) for data collection on crystals of several proteins. In this system data were processed

simultaneously with data collection. These results were also quite satisfactory.

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Thermal Motion Analysis in Tetraphenylmethane: a Lattice-Dynamical Approach

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Abstract

A 'non-rigid' harmonic lattice-dynamical model has been used for analyzing thermal motion in crystals of tetraphenylmethane. Empirical internal and external force fields which were derived for a series of

other aromatic hydrocarbons have been employed. The agreement between the calculated anisotropic temperature factors and the corresponding experimental values is excellent. Bond-length corrections for the general case of non-rigid thermal motion are obtained: the average C-C bond length in the phenyl

rings (excluding the bonds which are adjacent to the central C atom) is 1.395 Å, a value which is very close to spectroscopic or theoretical results.

Introduction

The problem of applying corrections for thermal libration to molecular geometry, as derived from X-ray or neutron diffraction data at room temperature, is becoming more and more pressing. This is because on one hand the accuracy of data collection has been considerably improved in recent times, and the e.s.d. of bond lengths is often in the range of 0.004 Å or less; on the other hand, a comparison with *ab initio* calculations or other techniques, such as microwave spectroscopy determinations (see for instance Boggs, Pang & Pulay, 1982) needs very accurate data to be significant.

In many instances, a rigid-body Schomaker & Trueblood (1968) treatment (here onwards ST) is sufficient for most purposes; however, for non-rigid molecules the problem becomes considerably more complex, and a general solution by only fitting temperature factors is not feasible, unless some information about the model is given (Busing & Levy, 1964; Johnson, 1970; Trueblood, 1978; He & Craven, 1985).

In our lattice-dynamical calculations on molecular crystals, we found that for a rigid molecule the evaluation of crystallographic thermal vibration parameters starting from the experimental molecular geometry and semiempirical potentials is not particularly difficult, at least when no extensive Coulombic interactions are present (Filippini, Gramaccioli, Simonetta & Suffritti, 1973, 1974*a,b*, 1976*a,b*, 1978).

A recent extension of our programme to non-rigid molecules does not involve a considerable increase of computing time or complexity in establishing the mathematical procedure: this happens if the normal coordinates of the isolated molecule are used as a basis for the calculations in the crystal (Bonadeo & Burgos, 1982; Gramaccioli, Filippini & Simonetta, 1982; Gramaccioli & Filippini, 1983, 1984*a,b*, 1985*a,b*; Filippini, Simonetta & Gramaccioli, 1984; Filippini, 1985).

For aromatic hydrocarbons, where acceptable semiempirical force fields are given in the literature, an obvious extension of our procedure might concern on one hand the larger condensed ring systems, which become more flexible on increasing the molecular dimensions, and on the other hand the polyphenyls. These extensions are important for verifying the general applicability of our routine, including the potentials, which should be widely transferable in order to be useful at least for a general-purpose crystallographic application. Among the few substances of this kind for which accurate data are available, we chose *o*-terphenyl (Gramaccioli & Filippini, 1985*b*) in the first place, and obtained a very good agreement

with the experimental **B**'s (obtained from neutron diffraction). This gave us a reasonable foundation to believe that our calculated coupling tensors

$$U(kk') = \langle \mathbf{u}(k)\mathbf{u}(k')^T \rangle$$

are near to 'reality', or they are at least good enough to be used for bond-length correction in the general case. In the equation given above, $\mathbf{u}(k)$ is the total displacement of the atom k from all the modes of vibration at any instant of time, and $\langle \dots \rangle$ denotes the time average [see, for instance, Willis & Pryor (1975)].

For many other polyphenyls, where good experimental data are available, our calculations cannot be safely applied, because there is evidence for substantial structural disorder or strongly anharmonic motion.

On the other hand, tetraphenylmethane is quite interesting, because: (1) there is a particularly reliable set of three structure determinations, with a discussion about the accuracy of the results, including the **B**'s (Robbins, Jeffrey, Chesick, Donohue, Cotton, Frenz & Murillo, 1975); (2) the molecular model is more complex than for *o*-terphenyl, and it gives a more extensive check of our treatment of non-rigid bodies; (3) good experimental spectroscopic results (IR and Raman) are available (Schlotter & Hudson, 1982, 1986) for testing the 'goodness' of the empirical force field.

Tetraphenylmethane forms tetragonal crystals, space group $P4_2c$, with molecular site symmetry 4 ($= S_4$), and unit-cell parameters $a = 10.896$ and $c = 7.280$ Å.

Method of calculation

Our procedure has already been reported (Gramaccioli & Filippini, 1983, 1985*a,b*, here onwards GF), and details about the Brillouin zone sampling are given in Filippini *et al.* (1976*a,b*). The van der Waals field is taken from Williams (1967), with a maximum interaction distance of 5.5 Å; the same parameters have been used for taking the internal van der Waals repulsion also into account. The internal (valence) force field proposed by Neto, Scrocco & Califano (1966) has been used for the 'in-plane' motion of the phenyl rings: for the 'out-of-plane' motion the force field proposed by Filippini, Simonetta & Gramaccioli (1984) has been used. Such force fields give a satisfactory agreement with the measured vibration frequencies in crystals of several aromatic hydrocarbons; they give acceptable results even for the diphenyl molecule, and are the same we used in calculating temperature factors in *o*-terphenyl, with no additional modifications to fit any specific tetraphenylmethane data. Because X-ray diffraction does not yield precise nuclear positions for H atoms, and the potentials are calibrated on a longer C-H distance than the average coming from X-ray structural determinations, the

Table 1. *Calculated and observed vibration frequencies (cm⁻¹), and symmetry species (first column) for tetraphenylmethane crystals at the Γ point*

The first and second columns of our results refer to assuming a C-H bond distance of 1.09 Å, or the corresponding 'X-ray' experimental value, respectively. The other columns are taken from Schlotter & Hudson (1982, 1986).

	This work		SH	
	(1)	(2)	obs	calc
B	34	30	39	38
E	39	36	40	42
A	44	37	48	46
A	51	47	—	56
E	62	56	54	63
E	68	60	67	71
A	79	71	—	77
E	83	76	83	80
A	91	81	—	79
B	92	78	84	92
E	98	88	104	98
B	98	89	—	96
B	99	96	105	105
B	106	100	—	113
A	118	108	—	122
E	123	115	121	124
B	135	126	132	135
A	146	128	135	138
E	150	133	143	144
B	158	143	154	150

position of hydrogen atoms has been recalculated by extending the C-H bond length to 1.09 Å, as in our previous works. The influence of such operation is considerable on the calculated frequencies (see Table 1).

As shown in GF, our calculations on the crystal include a number of internal degrees of freedom in the dynamical matrices, corresponding to those normal coordinates of the free molecule whose frequencies are below a certain value (in this case 200 cm⁻¹). Here, such a limit corresponds to including nine internal degrees of freedom to mix with the lattice modes; the contribution of higher frequency modes has been added as calculated for the free molecule (see GF and Gramaccioli *et al.*, 1982).

Discussion

The calculated frequencies at the Γ point ($\mathbf{k} = 0$) are reported in Table 1. These are in very good agreement with the observed values (Schlotter & Hudson, 1982), and with the calculations performed by the same authors (1986), who use a different field from ours. Such an agreement gives considerable support to the transferability of the empirical field we used for aromatic hydrocarbons (see above).

The first 15 × 15 block of the mean-square displacement tensor \mathbf{W} is reported in Table 2; the relationships of such a tensor to the ST molecular vibration tensors \mathbf{T} , \mathbf{L} and \mathbf{S} are given in GF. Owing to the high site symmetry ($\bar{4} = S_4$), we have a particularly good example for showing how the symmetry requirements for \mathbf{W} work: in fact, many components are zero, and this happens according to our first rule (see GF), *i.e.*

when the irreducible representations of the corresponding normal coordinates are different. When the representations are two-dimensional (the assignment of the symmetry species is given in the first line), the situation is more interesting: the blocks corresponding to the cross terms between symmetry-related normal coordinates (as, for instance, the blocks on the main diagonal in connection with E symmetry species) are diagonal, with equal elements (see our second rule in GF). However, the blocks corresponding to cross terms between normal coordinates with the same symmetry species E , but not related to each other by symmetry operations, are not diagonal. The inspection of the 13th and 14th columns of \mathbf{W} (which is relative to the last E symmetry species here considered) shows a number of interesting situations; we have 2 × 2 blocks with equal diagonal elements, and zero off-diagonal elements, or blocks with equal diagonal elements and non-zero opposite off-diagonal elements, or blocks with opposite diagonal elements and equal off-diagonal elements. Such situations arise because the representations are not the same, although they are equivalent (see GF, 1985*b*).

Apart from these symmetry considerations, the components of \mathbf{W} due to the internal degrees of freedom are clearly not negligible, and the mutual coupling, or the coupling between internal and translational or rotational motion are also not negligible; on the whole, however, the contribution of the rotational and translational modes is dominant.

The lattice-dynamical estimates of the ST molecular vibration tensors \mathbf{T} , \mathbf{L} and \mathbf{S} are reported in Table 3, along with the corresponding values derived from an ST fit to the whole molecule (only carbon atoms). The ST estimates are consistently higher than the values derived from lattice dynamics. A similar case was encountered also for *o*-terphenyl, and the situation can be easily explained, since in the ST fit the internal mode contribution is bound to be interpreted in terms of rigid-body translation and rotation. Such differences are not negligible when bond-length corrections are calculated.

The calculated anisotropic temperature factors (\mathbf{B} 's) for the carbon atoms are given in Table 4: the fit to the experimental values is excellent ($R = 13\%$). Also the averages of the observed and of the calculated values are not out of scale $\{\Delta B = \sum [B_{ij(\text{obs})} - B_{ij(\text{calc})}] / \sum B_{ij(\text{obs})} = 3\%\}$, a point which seems to exclude a strong TDS effect upon the collected intensity data. For the hydrogen atoms, the calculated temperature factors (isotropic) are given in Table 5: they also are in fair agreement with the experimental values, a remarkable point, since the latter are derived from X-ray diffraction. In Fig. 1, the calculated thermal ellipsoids (at 50% probability level) for the carbon atoms in the molecule are represented; in Fig. 2, the corresponding internal mode contribution is drawn on the same scale.

Table 2. *The first 15 × 15 block of the tensor W for tetraphenylmethane (atomic mass units × Å²)*

The reference system is Cartesian, coinciding with the crystal axes (and with the principal axes of inertia of the free molecule). The rows and columns refer, in sequence, to translational, rotational, and internal coordinates, in order of increasing frequency (cm⁻¹) in the isolated molecule, whose conformation is assumed to be the same as in the crystal. All values have been multiplied by 1000.

E		B	E		A	A	B	E		A	B	E		B
8059	0	0	-982	-642	0	0	0	-151	-300	0	0	246	-48	0
	8059	0	-642	982	0	0	0	-300	151	0	0	48	246	0
		8212	0	0	0	0	0	0	0	0	0	0	0	0
			2759	0	0	0	0	95	-253	0	0	201	86	0
				2759	0	0	0	253	95	0	0	86	-201	0
					3150	0	0	0	0	0	0	0	0	0
						1175	0	0	0	-226	0	0	0	0
							1124	0	0	0	29	0	0	15
								914	0	0	0	-31	-139	0
									914	0	0	-139	31	0
										690	0	0	0	0
											735	0	0	40
												533	0	0
													533	0
														389

Table 3. *Rigid-body vibrational tensors T, L and S (× 10⁴) for the tetraphenylmethane molecule (Å², rad², Å rad, respectively)*

(1) from our lattice-dynamical calculations; (2) from the ST fit to the experimental ('West Pa') data in Robbins *et al.* (1975).

	T			L			S		
(1)	256	0	0	12	0	0	12	7	0
		256	0	12	0	7	-12	0	0
			266	15	0	0	0	0	0
(2)	346	0	0	16	0	0	12	15	0
		346	0	16	0	15	-12	0	0
			293	24	0	0	0	0	0

Table 4. *Temperature factors (Å²) for carbon atoms in tetraphenylmethane in the form T = exp(-10⁻⁴B_{ij}h_ih_j)*

For each atom, the first line reports the observed values ('West Pa' data in Robbins *et al.*, 1975); the second reports the results of our lattice-dynamical estimates.

	B ₁₁	B ₂₂	B ₃₃	B ₁₂	B ₁₃	B ₂₃
C(0)	51 (1)	51 (1)	115 (3)	0	0	0
	53	53	112	0	0	0
C(1)	56 (1)	60 (1)	111 (3)	-5 (1)	10 (2)	0 (2)
	55	58	122	-2	0	-5
C(2)	71 (1)	83 (2)	129 (4)	-5 (1)	-4 (2)	-6 (2)
	62	73	134	-6	-8	-7
C(3)	83 (2)	111 (2)	134 (4)	-25 (2)	1 (2)	-20 (2)
	81	86	155	-19	-8	-23
C(4)	111 (2)	86 (2)	175 (4)	-26 (2)	23 (1)	-42 (3)
	104	77	194	-15	1	-40
C(5)	107 (2)	67 (2)	195 (4)	3 (2)	23 (3)	-21 (2)
	103	63	206	5	1	-31
C(6)	76 (2)	65 (1)	144 (4)	3 (1)	4 (2)	-11 (2)
	74	59	158	7	-2	-14

The corrected C-C bond-length values according to the general motion model (Scheringer, 1972*a,b*; Johnson, 1980) are reported in Table 6. The corrected values for the C-H bond lengths are not reported here, because it is difficult to establish atomic coordinates of the H atoms which are significant for our purposes when no neutron diffraction data are dealt with; moreover, anharmonicity of the stretching vibration does not seem to be negligible for such corrections (Craven & Swaminathan, 1985).

Table 5. *Isotropic temperature factors (Å²) for the hydrogen atoms in tetraphenylmethane*

The first column refers to observed values (same source as in Table 4), the second to our calculations.

	B _{obs}	B _{cal}
H(2)	3.4 (4)	4.6
H(3)	4.7 (4)	5.6
H(4)	5.0 (5)	6.2
H(5)	4.6 (5)	6.1
H(6)	4.3 (5)	4.6

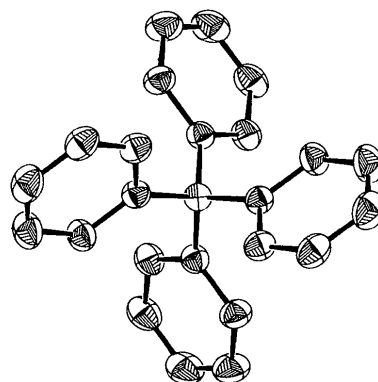


Fig. 1. Ellipsoids drawn at 50% probability level, representing temperature factors in tetraphenylmethane.

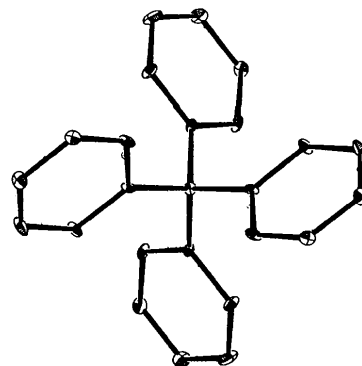


Fig. 2. The contribution of the internal modes to the thermal motion ellipsoids in Fig. 1, drawn at the same scale.

Table 6. Bond distances (Å) in tetraphenylmethane

For each bond, the columns show, in sequence: (1) the uncorrected bond length;* (2) the corrected bond length according to a rigid body ST fit to the temperature factors; (3) the corrected bond length for a 'riding' motion; (4) the corrected bond length for the general case of a non-rigid molecule using the results of our lattice-dynamical estimations.

	(1)	(2)	(3)	(4)
C(0)–C(1)	1.551	1.554	1.553	1.557
C(1)–C(2)	1.403	1.406	1.412	1.411
C(1)–C(6)	1.389	1.392	1.397	1.399
C(2)–C(3)	1.385	1.387	1.393	1.394
C(3)–C(4)	1.381	1.384	1.386	1.392
C(4)–C(5)	1.384	1.387	1.397	1.394
C(5)–C(6)	1.392	1.395	1.403	1.401
Average in the ring	1.3890	1.3918	1.3963	1.3985

* Data from the 'West Pa' set in Robbins *et al.* (1975).

In Tables 1–6, for reasons of space, only one set of experimental data ['West Pa' in Robbins *et al.* (1975)] is reported, and the calculated data have been obtained starting from the corresponding unit cell and atomic coordinates. For sake of completeness, the results of the other two sets are reported in Tables 1*a* to 6*a*;^{*} there is, however, no substantial difference among the various sets in the results concerning the lattice-dynamical estimations.

The libration corrections which have been derived from lattice dynamics are in general larger than from either the 'rigid-body' ST fit or the 'riding' model. The average C–C bond length in the phenyl ring is 1.398, 1.393, and 1.397 Å, respectively, for the three experimental sets ('West Pa', 'Texas', and 'East Pa'). If the shorter value ('Texas') is excluded, because of possible systematic error in the unit-cell determination [in line with Robbins *et al.* (1975)], then the average of the corrected values becomes very close to the average C–C bond length in toluene (1.398 Å), as determined by gas-phase electron diffraction (Seip, Schultz, Hargittai & Szabó, 1977). The average of the C–C bonds in the ring which are not adjacent to the central C atom (1.395 Å) also becomes very close to the value for benzene (1.397 Å), and to the corresponding average (1.396 Å) of microwave determinations in monosubstituted benzene derivatives. These values are practically independent of the nature of the substituents [for an exhaustive reference list, see Domenicano, Schultz, Kolonits & Hargittai (1979)].

Therefore, tetraphenylmethane is not only a good example of agreement between different X-ray crystal structure determinations, but it is also a striking example of how the results of spectroscopic and crystallographic investigations can be mutually consistent and helpful.

* Tables 1*a*–6*a* have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43138 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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