Crystal Structure and ^{35}Cl NQR of (−)β-(trichloromethyl)-β-propiolactone. Comparison with (±)β-(trichloromethyl)-β-propiolactone

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The crystal structure of (−)β-(trichloromethyl)-β-propiolactone at room temperature is reported, as is the ^{35}Cl NQR spectrum in the range $77 \leqq T/K \leqq 323.5$. The compound crystallizes with the space group D_2^4–$2_1 2_1 2_1$, $Z = 8$, $a = 2416.0$ (10) pm, $b = 975.6$ (4) pm, $c = 595.0$ (2) pm. The intramolecular distances and angles of the two crystallographically independent (–) molecules in the unit cell are equal within the limits of error. The spread of the ^{35}Cl NQR spectrum is within 600 kHz, not changing in the temperature range covered. The crystal structure and ^{35}Cl NQR spectrum are discussed.

The results found for the (–) compound are compared with the corresponding ones reported for the (±) compound [1], and the influence of the different intramolecular interactions in the two solid states of the chemically identical compounds on the NQR spectrum is discussed.

Introduction

Recently we have reported the crystal structure of (±)β-(trichloromethyl)-β-propiolactone and the ^{35}Cl NQR spectrum of it for $T \geqq 77$ K [1]. Besides the interest on the dynamical behavior of the group $-CCl_3$ as a function of the bond $R-CCl_3$ and its environment in the crystal lattice, see e.g. [2], we are engaged in studies of the crystal field by nuclear quadrupole resonance, NQR [3].

It was pointed out that molecules with one or more optically active centers offer an interesting possibility for the study of van der Waals interactions in molecular solids [4]. In first approximation one may assume unchanged intramolecular bond properties of the molecule in the solid, whether it is the (−) molecule (the (+) one, respectively) or it is the (±) molecule pair. Differences in the NQR spectra of (−) and (±) should be solely due to that part of the electric field gradient tensor, EFGT, (Φ_{XX}, Φ_{YY}, Φ_{ZZ}) which is caused by the crystal field. In the following, we report the crystal structure and the ^{35}Cl NQR spectrum of (−)β-(trichloromethyl)-β-propiolactone and compare the results with the ones found for the corresponding (±) compound [1].

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Experimental

As in case of the (±) compound, colorless needles were grown form the solution of commercial material (Aldrich) for the X-ray diffraction work. Polycrystalline material was used for the ^{35}Cl NQR studies. X-ray diffraction intensities were collected on a Stoe-Stadi-4 four circle diffractometer. After appropriate correction of the intensity data for absorption and Lorentz-polarization factor, the crystal structure was solved by direct methods [5] and from Fourier synthesis. All atoms could be located. The structure parameters were refined by means of the full matrix least squares method [6]. The positions of the hydrogen atoms were found from difference Fourier maps and refined with fixed isotropic temperature factors.

The ^{35}Cl NQR spectra were registered as a function of temperature T with the setup described in [1]. Errors in T and ν (^{35}Cl) measurement are the same as given for the (±) compound [1].

Results

In Table 1 the crystallographic data, lattice constants, etc. of (−)β-(trichloromethyl)-β-propiolactone are listed, together with the experimental conditions for the X-ray diffraction. The compound crystallizes within the acentric space group D_2^4–$P2_1 2_1 2_1$, $a =$ 2416.0 (10) pm, $b = 975.6$ (4) pm, $c = 595.0$ (2) pm, $Z = 8$

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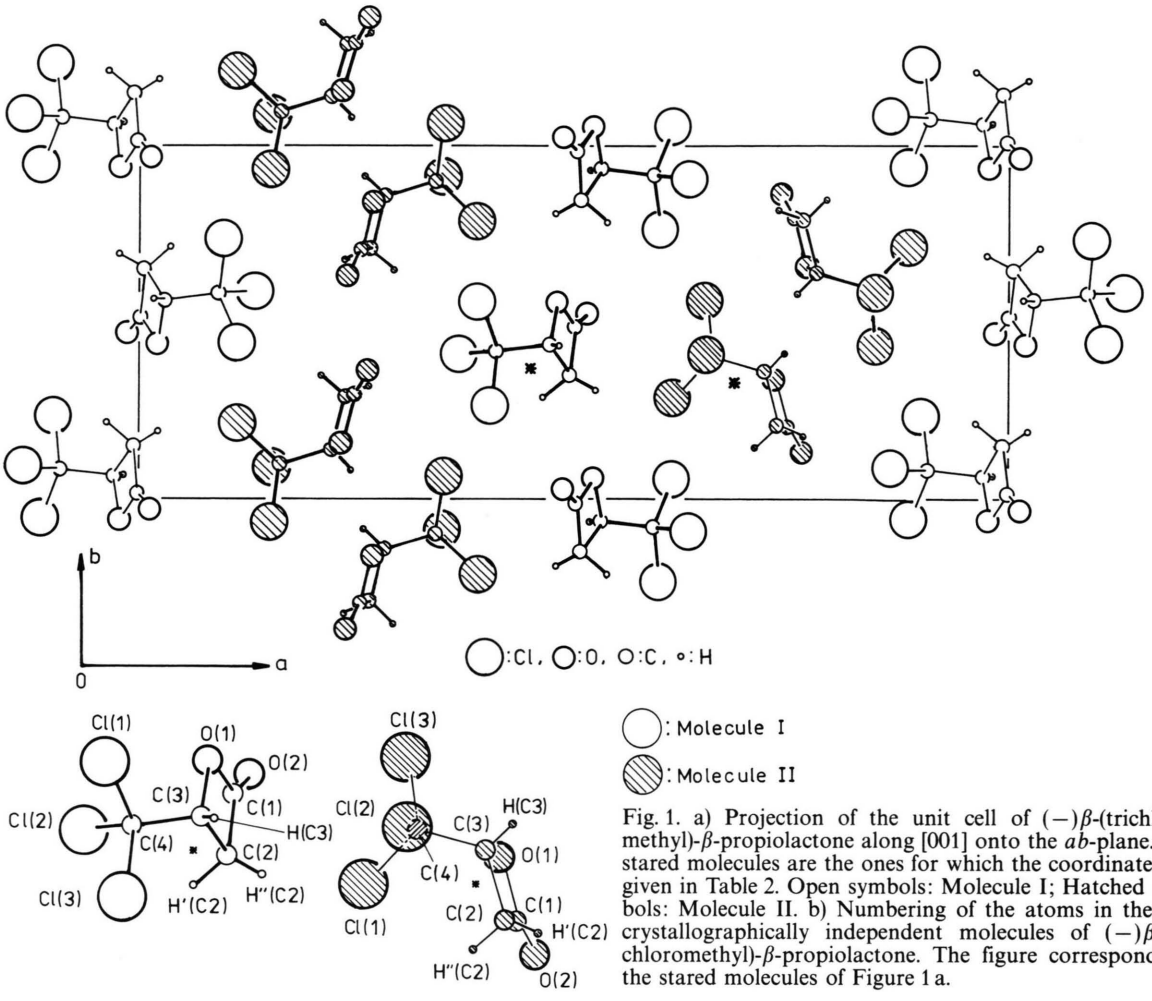

Fig. 1. a) Projection of the unit cell of (−)β-(trichloromethyl)-β-propiolactone along [001] onto the *ab*-plane. The stared molecules are the ones for which the coordinates are given in Table 2. Open symbols: Molecule I; Hatched symbols: Molecule II. b) Numbering of the atoms in the two crystallographically independent molecules of (−)β-(trichloromethyl)-β-propiolactone. The figure corresponds to the stared molecules of Figure 1 a.

molecules in the unit cell. Consequently, there are two molecules, I and II, in the asymmetric unit of the elementary cell.

Figure 1 a shows the projection of the unit cell along [001] onto the *ab* plane, and in Fig. 1 b the numbering of the atoms within the molecules I and II is given. The numbering of the atoms in the report on the structure of the (±) compound [1] is in agreement with that used here. Positional and thermal parameters of the atoms are listed in Table 2; in Table 3 intra- and intermolecular distances are given, together with the corresponding angles. For further information on the structure determination see [10].

The ^{35}Cl NQR results are plotted in Fig. 2, $\nu(^{35}Cl) = f(T)$. The data of the ^{35}Cl NQR experiments are rationalized by parameterizing the curves in Fig. 2 according to

$$\nu(^{35}Cl) = \sum_{i=-1}^{2} a_i T^i. \quad (1)$$

In Table 4 one finds the coefficients of (1) for the title compound; there the resonance frequencies at two selected temperatures, 77 K and 295.3 K, are also given.

Discussion

Structure and ^{35}Cl NQR of (−) β-(trichloromethyl)-β-propiolactone

The ^{35}Cl NQR frequencies decrease smoothly with increasing temperature *T*. This is expected from the

Table 1. Experimental conditions for the crystal structure determination and crystallographic data of (−)β-(trichloromethyl)-β-propiolactone. Diffractometer: Stoe-Stadi 4; wavelength: 71.069 pm (MoKα); monochromator: Graphite (002); scan: $2\vartheta/\omega$.

Formula (Molar mass)	$C_4H_3Cl_3O_2$ (189.43)
Crystal size, habitus	$(0.15 \times 0.2 \times 2.5)$ mm^3, needle
Colour	Colourless
Temperature/K	294
Absorption coefficient (μ/m^{-1})	1232.9
$(\sin\vartheta/\lambda)_{max}$/ pm	0.005385
Number of measured reflexions	2571
Symmetry independent reflexions	1820
Reflexions considered	1748
Number of free parameters	180
F (000)	752
$R(F)$	0.044
$R_w(F)$	0.039
Lattice constants:	
a/pm	2416.0 (10)
b/pm	975.6 (4)
c/pm	595.0 (2)
Volume of the unit cell V 10^{-6}/(pm)^3	1402.44 (4)
Space group	$P2_12_12_1$-D_2^4
Formula units per unit cell	8
ϱ_{calc}/Mg m^{-3}	1.794
ϱ_{pykn}/Mg m^{-3}	1.83 ($T = 299$ K)
Point positions	All atoms in 4a [7]
	$x, y, z;\ \frac{1}{2}-x, \bar{y}, \frac{1}{2}+z$
	$\frac{1}{2}+x, \frac{1}{2}-y, \bar{z};\ \bar{x}, \frac{1}{2}+y, \frac{1}{2}-z.$

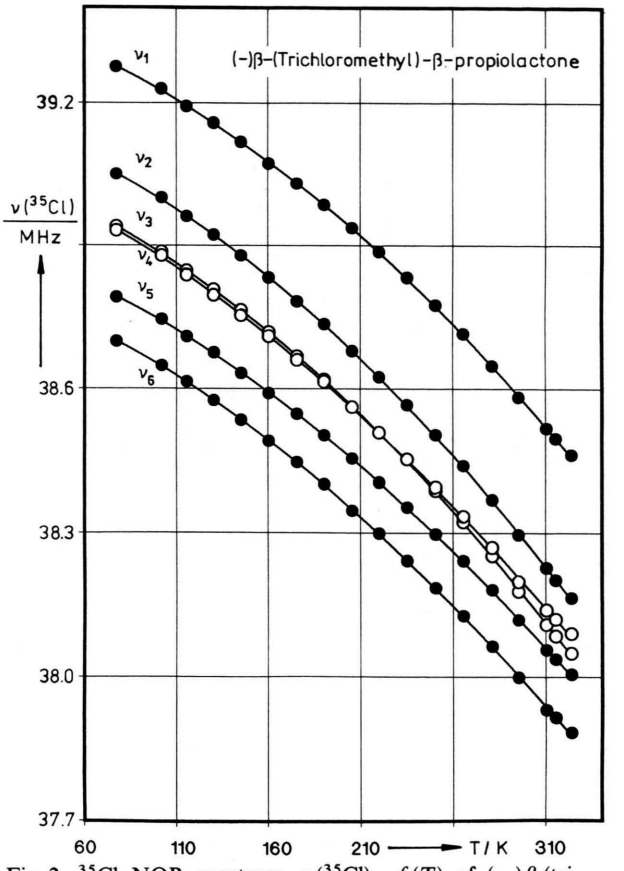

Fig. 2. ^{35}Cl NQR spectrum, $\nu(^{35}Cl) = f(T)$ of (−)β-(trichloromethyl)-β-propiolactone.

dynamics of a molecule in the lattice where the librational motions increase with increasing T, averaging the EFGT and thereby lowering the NQR frequencies, in agreement with the predictions of Bayer [8]. The slope of the curves $\nu(^{35}Cl) = f(T)$ behaves similar for the six observed frequencies, and the spread of them is not changing much; $T = 77$ K: $\nu_{max} - \nu_{min} = 576$ kHz; $T = 295$ K: $\nu_{max} - \nu_{min} = 584$ kHz.

Three carbon-carbon bonds are in the molecule, see Figure 1 b. Within the limits of error we find (all distances in pm) for molecule I and molecule II $\langle d(C(1)-C(2))\rangle = 151.0(9)$, $\langle d(C(2)-C(3))\rangle = 152.0(10)$, and $\langle d(C(3)-C(4))\rangle = 151.0(9)$; the distances carbon-carbon can be considered as equal. Also the distances carbon-chlorine are the same for the two crystallographically independent molecules with the mean values $\langle d(C(4)-Cl(1))\rangle = 175.2(6)$, $\langle d(C(4)-Cl(2))\rangle = 179.4(6)$, $\langle d(C(4)-Cl(3))\rangle = 175.5(6)$. Two of the three C–Cl bond distances do not differ whereas the third one, $d(C(4)-Cl(2))$, is stretched by 4 pm in both molecules, I and II.

The distances C(1)–O(1) are 136.5 (6) and 138.1 (8), respectively, about 10 pm shorter than $\langle d(C(3)-O(1))\rangle = 147.3(3)$. This is understandable, because C(1) is the carbon atom of the keto group C(1) = O(2), which shortens the distance C(1)–O(1) compared to C(3)–O(1). The distances C(1)–O(2) are in the mean 118.0 (7) pm. Whereas the molecules I and II do not differ in the bond lengths within the limits of error, there are differences in the bond angles which are considerably above these limits. This shows that bond distances are hard, bond angles weak bond parameters, see also Table 3.

The ring C(1)–C(2)–C(3)–O(1) is for molecule I and molecule II nearly planar. We have calculated the best plane through the four ring atoms and find

Molecule I:

$$22.0975\,x + 1.0634\,y + 2.3164\,z = 12.5655; \quad (2)$$

Table 2. Positional and thermal parameters of (−)β-(trichloromethyl)-β-propiolactone, $C_4H_3Cl_3O_2$; the temperature factor is of the form

$$T = \exp\{-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)\}.$$

The U_{ij} are given in $(pm)^2$; U is isotropic mean for the hydrogen atoms.

Atom	x/a	y/b	z/c	$U_{11}(U)$	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Molecule I									
Cl(1)	0.3878(1)	0.5585(2)	0.9714(3)	792(11)	479(10)	539(11)	67(9)	175(12)	−110(10)
Cl(2)	0.3670(1)	0.4109(2)	0.5610(3)	556(9)	499(10)	481(10)	−70(8)	−97(9)	21(11)
Cl(3)	0.4024(1)	0.2651(2)	0.9592(3)	936(14)	458(10)	516(11)	48(10)	79(11)	174(10)
O(1)	0.4783(2)	0.5609(4)	0.6103(7)	546(25)	446(27)	457(28)	−118(24)	43(25)	6(26)
O(2)	0.5146(2)	0.5290(6)	0.2619(9)	669(33)	919(42)	411(30)	20(31)	84(28)	49(31)
C(1)	0.4990(2)	0.4856(8)	0.4361(0)	426(39)	691(52)	385(44)	7(35)	21(36)	−125(47)
C(2)	0.4939(3)	0.3496(7)	0.5571(15)	584(44)	489(45)	552(52)	176(34)	143(43)	−22(44)
C(3)	0.4693(3)	0.4357(9)	0.7430(12)	534(49)	535(44)	379(45)	−18(39)	−86(35)	−47(43)
C(4)	0.4093(2)	0.4188(6)	0.8088(10)	514(36)	328(34)	379(36)	−6(32)	−41(31)	19(34)
H′(C2)	0.4693(20)	0.2765(56)	0.5124(116)	600					
H″(C2)	0.5265(21)	0.2956(55)	0.5946(108)	600					
H(C3)	0.4862(28)	0.4402(85)	0.8233(116)	600					
Molecule II									
Cl(1)	0.6121(1)	0.2873(2)	0.0341(3)	536(9)	492(10)	580(11)	−49(8)	−78(11)	−97(10)
Cl(2)	0.6516(1)	0.4131(2)	0.4426(3)	749(11)	621(11)	319(9)	−41(10)	11(10)	−28(10)
Cl(3)	0.6506(1)	0.5659(2)	0.0268(3)	878(12)	367(9)	634(12)	130(9)	94(12)	121(11)
O(1)	0.7317(2)	0.3416(5)	−0.1321(7)	619(29)	524(31)	370(26)	22(26)	117(25)	27(24)
O(2)	0.7627(2)	0.1313(6)	−0.2429(9)	925(42)	784(42)	808(39)	105(34)	167(37)	−299(35)
C(1)	0.7459(3)	0.2058(8)	−0.1023(13)	545(44)	468(51)	623(54)	19(37)	6(42)	−104(44)
C(2)	0.7345(3)	0.2061(8)	0.1465(13)	586(48)	474(49)	501(47)	182(40)	35(41)	94(40)
C(3)	0.7195(3)	0.3568(7)	0.1097(12)	550(42)	388(42)	474(46)	−50(35)	−55(38)	−12(36)
C(4)	0.6608(2)	0.4027(6)	0.1439(10)	558(38)	331(36)	334(33)	57(34)	61(33)	7(32)
H′(C2)	0.7535(25)	0.1635(69)	0.2529(104)	600					
H″(C2)	0.7019(20)	0.1345(57)	0.1849(97)	600					
H(C3)	0.7419(23)	0.4171(53)	0.1694(100)	600					

Table 3. Crystal structure of (−)β-(trichloromethyl)-β-propiolactone, $C_4H_3Cl_3O_2$; Intra- and intermolecular distances, (d/pm), and angles (°).

Atoms	d/pm		Atoms	Angle/°	
	I	II		I	II
Cl(1)–C(4)	175.0(6)	175.3(6)	Cl(1)–C(4)–C(3)	110.0(5)	113.0(4)
Cl(2)–C(4)	179.5(6)	179.4(6)	Cl(2)–C(4)–C(3)	109.7(4)	105.5(5)
Cl(3)–C(4)	175.4(6)	175.5(6)	Cl(3)–C(4)–C(3)	108.4(5)	110.4(5)
C(1)–C(2)	151.5(9)	150.6(10)	Cl(1)–C(4)–Cl(2)	108.6(3)	108.9(3)
C(1)–O(1)	136.5(6)	138.1(8)	Cl(1)–C(4)–Cl(3)	110.9(3)	109.9(3)
C(1)–O(2)	118.1(6)	118.0(8)	Cl(2)–C(4)–Cl(3)	109.2(3)	109.0(3)
C(2)–C(3)	151.1(11)	153.0(9)	O(1)–C(1)–O(2)	126.2(7)	125.8(8)
C(3)–C(4)	151.1(9)	150.2(9)	O(1)–C(1)–C(2)	94.6(4)	94.5(6)
C(3)–O(1)	147.1(7)	147.6(7)	O(2)–C(1)–C(2)	139.2(7)	139.6(9)
			C(1)–C(2)–C(3)	83.8(8)	84.5(6)
$Cl(1^I)\cdots Cl(1^{II})$ (i)	369.3(3)		C(2)–C(3)–C(4)	120.5(6)	119.4(6)
$Cl(1^I)\cdots Cl(2^{II})$ (i)	362.5(3)		O(1)–C(3)–C(4)	111.8(6)	110.6(6)
$Cl(2^I)\cdots Cl(1^{II})$ (ii)	375.0(3)		O(1)–C(3)–C(2)	90.6(6)	89.7(5)
$Cl(2^I)\cdots Cl(3^{II})$ (iii)	342.3(3)		C(1)–O(1)–C(3)	90.9(5)	91.2(5)
$Cl(3^I)\cdots Cl(2^{II})$ (iv)	372.0(3)				
$Cl(3^I)\cdots Cl(3^{II})$ (iii)	371.2(4)				
$Cl(2^I)\cdots O(2^{II})$ (v)	317.9(3)				
$Cl(3^I)\cdots O(2^I)$ (vii)	347.6(3)				
$Cl(1^{II})\cdots O(1^I)$ (iii)	322.3(4)				
$Cl(3^{II})\cdots O(1^{II})$ (vi)	360.7(4)				

i: $1-x, \frac{1}{2}+y, \frac{3}{2}-z$ ii: $1-x, \frac{1}{2}+y, \frac{1}{2}-z$ iii: $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$ vii: $1-x, y-\frac{1}{2}, \frac{3}{2}-z$

iv: $1-x, -\frac{1}{2}+y, \frac{3}{2}-z$ v: $-\frac{1}{2}+x, \frac{1}{2}-y, -z$ vi: $\frac{3}{2}-x, 1-y, \frac{1}{2}+z$

Table 4: Power series expansion of $\nu(^{35}Cl) = f(T)$ for (−)β-(trichloromethyl)-β-propiolactone. $f(T) = \sum_{i=-1}^{2} a_i T^i$; the temperature range within which the least squares adjustment is valid is for $\nu_1 \ldots \nu_6$: 77 K − 323.5 K; z = Number of data; σ = Standard deviations.

No	$\nu(^{35}Cl)\left(\frac{T}{K}\right)$ MHz	$\nu(^{35}Cl)\left(\frac{T}{K}\right)$ MHz	z	σ kHz	a_{-1} MHz · K	a_0 MHz	$a_1 \cdot 10^3$ MHz · K^{-1}	$a_2 \cdot 10^6$ MHz · K^{-2}
ν_1	39.276 (77)	38.584 (295.3)	18	2.4	0.496	39.381	−0.982	−5.790
ν_2	39.053 (77)	38.296 (295.3)	18	2.6	0.215	39.181	−1.205	−6.014
ν_3	38.941 (77)	38.180 (295.3)	18	2.8	0.967	39.054	−1.143	−6.137
ν_4	38.933 (77)	38.200 (295.3)	18	3.6	−1.539	39.116	−1.773	−4.338
ν_5	38.793 (77)	38.120 (295.3)	18	2.2	−1.396	38.951	−1.452	−4.537
ν_6	38.700 (77)	38.000 (295.3)	18	3.1	−2.033	38.875	−1.559	−4.634

The signal to noise ratio (Lock-in technique, recorder, time constant 10 s) is 23 to 30 at 77 K and decreases to ≈4 near the melting point of the compound.

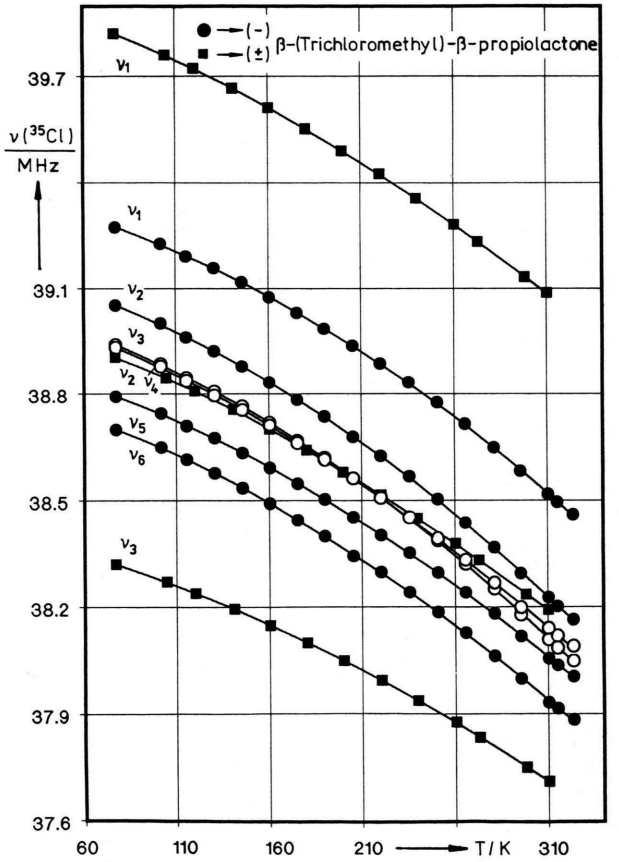

Fig. 3. Combined spectrum $\nu(^{35}Cl) = f(T)$ of (−)- and (±)β-(trichloromethyl)-β-propiolactone.

Molecule II:

$$22.9320\,x + 2.5728\,y + 1.0244\,z = 17.5271. \quad (3)$$

The deviations of the plane-atoms from the best plane are small, ≦1.3 pm (molecule I) and ≦0.3 pm (molecule II). The carbonyl oxygen O(2) is slightly out of plane, −2.5 pm (molecule I) and 5.2 pm (molecule II).

On comparing the distances C−Cl with the ^{35}Cl NQR frequencies it shows up that the crystal field effect is as strong as the bond relation $\Phi_{ZZ} = a(1/d^3)$ contribution [9] and an assignment of the frequencies to the six different Cl-positions is, without single crystal NQR studies, not possible.

Comparison of (−)β-(trichloromethyl)-β-propiolactone and (±)β-(trichloromethyl)-β-propiolactone

Firstly we consider the molecular structure of the two solids. In Table 5 we have listed the mean intra- and intermolecular distances and angles of the molecules I and II of the (−) crystal together with the corresponding data of the (±) crystal, given in [1]. Within the limits of the error in the structure determination of the (−) compound there is no severe difference in the intramolecular distances. Also the intermolecular distances show no strong differences. The deviations of the ring atoms from the best plane through C(1)−C(2)−C(3)−O(1) of the (±) compound are ≦1.4 pm (4.4 pm for O(2)). The plane equation for the ring atoms of (±)β-trichloromethyl-β-propiolactone is

$$7.9230\,x + 3.2488\,y + 1.8967\,z = 6.6531. \quad (4)$$

On the other hand, the crystal field effect in the ^{35}Cl NQR spectrum is much stronger for the (±) compound than for the (−) compound. This is seen in Figure 3. The overall splitting of the triplet spectrum of the (±) compound is by far larger than that of the (−) compound. It is interesting to note that, in simple approximation, one would expect weaker crystal fields

Table 5. Comparison of intra- and intermolecules distances d/pm in (−)β-(trichloromethyl)-β-propiolactone (molecule I and molecule II of the asymmetric unit) and in (±)β-(trichloromethyl)-β-propiolactone

Atoms	(−) d/pm		(±) d/pm
	I	II	
Cl(1)–C(4)	175.0(6)	175.3(6)	175.6(3)
Cl(2)–C(4)	179.5(6)	179.4(6)	177.6(3)
Cl(3)–C(4)	175.4(6)	175.5(6)	175.9(3)
C(1)–C(2)	151.5(9)	150.6(10)	150.4(4)
C(1)–O(1)	136.5(6)	138.1(8)	138.0(3)
C(1)–O(2)	118.1(8)	118.0(8)	117.7(3)
C(2)–C(3)	151.1(11)	153.9(9)	152.6(4)
C(3)–C(4)	151.1(9)	150.2(9)	151.5(3)
C(3)–O(1)	147.1(7)	147.6(7)	146.2(3)

	*			*	
$O(1)^{I} \cdots C(2)^{Ia}$	01	350.7	$O(2) \cdots C(3)^{a}$	20	343.6
$O(2)^{I} \cdots C(2)^{Ib}$	02	366.4	$O(2) \cdots C(3)^{b}$	21	353.7
$O(1)^{II} \cdots C(3)^{IIa}$	03	352.2	$O(2) \cdots O(2)^{a}$	22	340.7
$O(2)^{II} \cdots C(2)^{IIa}$	04	355.8	$O(2) \cdots O(1)^{a}$	23	324.2
$Cl(2)^{I} \cdots O(2)^{IIa}$	05	317.9	$Cl(3) \cdots O(2)^{b}$	24	314.5
$Cl(3)^{I} \cdots O(2)^{Ia}$	06	347.6	$Cl(3) \cdots O(2)^{c}$	25	355.1
$Cl(1)^{II} \cdots O(1)^{Ia}$	07	322.3	$Cl(3) \cdots O(2)^{d}$	26	367.8
$Cl(2)^{II} \cdots O(1)^{IIa}$	08	372.3			
$Cl(3)^{II} \cdots O(1)^{IIa}$	08	360.7			
$Cl(1)^{I} \cdots Cl(1)^{IIa}$	09	369.3	$Cl(1) \cdots Cl(2)^{a}$	27	372.2
$Cl(1)^{I} \cdots Cl(2)^{IIa}$	10	362.5	$Cl(1) \cdots Cl(1)^{a}$	28	367.4
$Cl(2)^{I} \cdots Cl(1)^{IIb}$	11	375.0	$Cl(2) \cdots Cl(2)^{a}$	27	371.2
$Cl(2)^{I} \cdots Cl(3)^{IIa}$	12	342.3			
$Cl(3)^{I} \cdots Cl(2)^{IIb}$	13	372.0			
$Cl(3)^{I} \cdots Cl(3)^{IIa}$	12	371.2			
$Cl(1)^{I} \cdots C(2)^{IIb}$	14	399.8	$Cl(3) \cdots C(1)^{a}$	29	380.8
$Cl(2)^{I} \cdots C(2)^{IIc}$	15	398.0	$Cl(3) \cdots C(2)^{a}$	30	359.2
$Cl(2)^{I} \cdots C(2)^{IId}$	16	381.8	$Cl(1) \cdots C(2)^{a}$	30	389.0
$Cl(2)^{I} \cdots C(1)^{IIc}$	17	397.3	$Cl(1) \cdots C(1)^{b}$	31	383.4
$Cl(3)^{I} \cdots C(1)^{IIb}$	18	388.6			
$Cl(3)^{I} \cdots C(1)^{Ia}$	19	367.4			
$Cl(1)^{II} \cdots C(1)^{Ia}$	19	398.8			
$Cl(2)^{II} \cdots C(3)^{IIa}$	03	396.4			

*

01: $1-x, \frac{1}{2}+y, \frac{3}{2}-z$
02: $1-x, \frac{1}{2}+y, \frac{1}{2}-z$
03: $\frac{3}{2}-x, 1-y, \frac{1}{2}-z$
04: $\frac{3}{2}-x, -y, \frac{1}{2}-z$
05: $x-\frac{1}{2}, \frac{1}{2}-y, -z$
06: $1-x, y-\frac{1}{2}, \frac{3}{2}-z$
07: $1-x, y-\frac{1}{2}, \frac{1}{2}-z$
08: $\frac{3}{2}-x, 1-y, z+\frac{1}{2}$
09: $1-x, \frac{1}{2}+y, \frac{3}{2}-z$
10: $1-x, \frac{1}{2}+y, \frac{3}{2}-z$
11: $1-x, \frac{1}{2}+y, \frac{1}{2}-z$
12: $1-x, y-\frac{1}{2}, \frac{1}{2}-z$
13: $1-x, y-\frac{1}{2}, \frac{3}{2}-z$
14: $1-x, \frac{1}{2}+y, \frac{3}{2}-z$
15: $1-x, \frac{1}{2}+y, \frac{1}{2}-z$

*

16: $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$
17: $1-x, \frac{1}{2}+y, \frac{1}{2}-z$
18: $x-\frac{1}{2}, \frac{1}{2}-y, -z$
19: $1-x, y-\frac{1}{2}, \frac{3}{2}-z$
20: $x, \frac{1}{2}-y, \frac{1}{2}+z$
21: $1-x, \frac{1}{2}+y, \frac{1}{2}-z$
22: $1-x, 1-y, 1-z$
23: $1-x, y-\frac{1}{2}, \frac{1}{2}+z$
24: $x, \frac{3}{2}-y, z-\frac{1}{2}$
25: $x, \frac{1}{2}-y, z-\frac{1}{2}$
26: $1-x, \frac{1}{2}+y, \frac{1}{2}-z$
27: $2-x, y-\frac{1}{2}, \frac{1}{2}-z$
28: $2-x, -y, -z$
29: $x, \frac{3}{2}-y, z-\frac{1}{2}$
30: $x, \frac{1}{2}-y, z-\frac{1}{2}$
31: $x, \frac{1}{2}-y, z-\frac{1}{2}$

in the (±) compound than in the (−) compound because in the (±) compound with its centrosymmetric space group dipole fields should compensate. The temperature dependence of the frequencies is nearly the same for both solid state arrangements.

So, we claim that the change in the intermolecular distances and thereby changes in the van der Waals interactions Cl ··· Cl, Cl ··· O, etc., are reflected by the changes in the observed electric field gradients Φ_{ZZ} [4]. In a further step, the assignment of the ^{35}Cl NQR frequencies to the crystallographic positions of the chlorines has to be done by Zeeman split NQR spectroscopy.

Finally, we wish to mention that also in the infrared spectrum we could observe the different symmetry of the two compounds from the splitting of the C=O band in the (−)β-(trichloromethyl)-β-propiolactone and also a shift of the center of gravity of this doublet compared to the C=O band of the (±) compound.

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[10] Further information on the crystal structure determination may be obtained from Fachinformationszentrum Karslruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-7514 Eggenstein-Leopoldshafen 2, Germany. Inquiries should be accompanied by the depository number CSD-56953, the names of the authors and the full literature reference.