

Quinuclidine-Thiourea Inclusion Compound. A Perfect van der Waals Cavity

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Abstract

Thiourea reacts with 1-azabicyclo[2.2.2]octane (quinuclidine, Q) in methanol at room temperature leading to the formation of Q(3-thiourea). Single crystal X-ray diffraction analysis shows that the product has a rhombohedral structure, $\alpha = \beta = 90^\circ$, $\gamma = 120^\circ$, $a = b = 15.9371(2)$, $c = 12.4248(2)$, which may be described as a thiourea matrix defining hexagonal channels where the quinuclidine molecules are located. The ^{13}C -cross polarisation magic angle spinning (CP-MAS) study indicates that the guest inside the cavities has high conformational, rotational and translational mobility at room temperature. Thermal studies indicate that the structural identity of the thiourea matrix remains after a partial loss of amine, and this result suggests that the thiourea-amine inclusion compound possesses an open host structure, or that a topotactic process is associated.

Introduction

It is known that thiourea forms crystalline compounds with a variety of organic species [1–4]. Many of these compounds may be described as inclusion compounds in which thiourea molecules define a structure containing linear, hexagonal channels where individual guest molecules are accommodated. Most available information deals with hydrocarbon-thiourea systems with cyclohexane, among others [5]. They have been extensively studied by various physical methods including solid-state NMR techniques, X-ray diffraction analysis and thermochemistry [6]. Information about systems containing guest species with functional groups like amines is, however, rather limited. In a previous communication we have reported on the synthesis of thiourea-amine inclusion compounds, pointing out that the more appropriate amines to form inclusion compounds are secondary amines with saturated bicyclic substituents [7]. Indeed, on the basis of X-ray diffraction analysis and composition data it was shown that these amines, in contrast with other bifunctional amines which lead to laminar products [8] (Figure 1), form conventional thiourea inclusion compounds [9]. In this paper we describe the complete characterisation, using single-crystal X-ray diffraction analysis, ^{13}C CP-MAS NMR spectroscopy, and thermal analysis, DTA and TG, of the title compound.

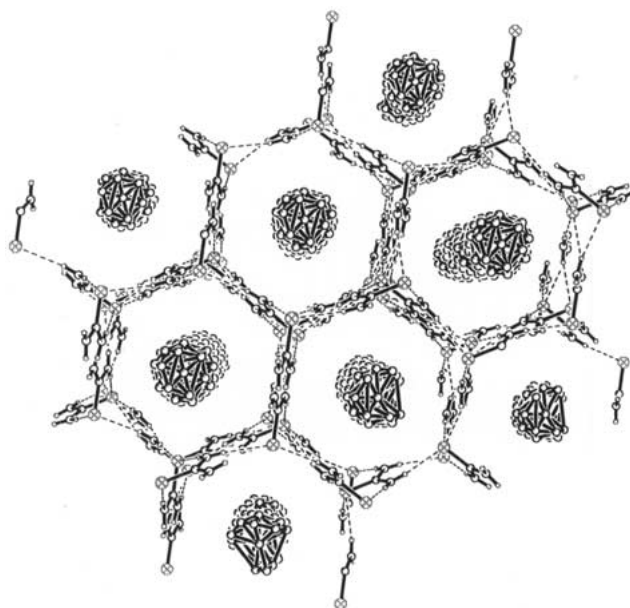


Figure 1. Structure of the compound Q(3 thiourea). View along the host hexagonal channels.

Experimental

Commercially available reagents were used as received. Crystals of the quinuclidine-thiourea inclusion compound were obtained by slow evaporation of the solvent at room temperature from solutions of both thiourea and the corresponding amine. The amine to thiourea molar ratios was always greater than the ratios expected for the products. After about 24 hours, beautiful crystals with well-formed

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faces may be separated. Hexagonal needles showing a characteristic behaviour under the polarized light microscope may be rapidly distinguished from pure thiourea, which crystallize as tetragonal prisms. The products were washed with cold methanol and dried under vacuum. The crystals decompose in water producing a characteristic visually observable dynamic effect [10]. Q(3 thiourea) was analyzed for C, H, N and S in a Perkin Elmer 240C, *anal.* Found. C 34.88, H 7.53, N 28.91, S 28.49, Calc. C 35.36, H 7.42, N 28.87, S 28.34. The ^{13}C CP-MAS NMR spectra were recorded on a Bruker MSL-100 spectrometer at a frequency of 100.63 MHz for ^{13}C . The number of scans was 400 with 5.5 μs 90° pulses; 1 m s cross-polarization contact time; 41 m s acquisition time during proton decoupling, and 5 s recycle delay. The polycrystalline powder sample was spun at a frequency of 4 kHz. The chemical shifts are given relative to tetramethylsilane (TMS) used as internal standard. DTA and TG were recorded on STA 409 Netzsch equipment, flow rate 5°/min and nitrogen atmosphere as purge gas. A single crystal was selected for the X-ray diffraction analysis. Data were collected on a Siemens R3m/V diffractometer with graphite monochromated Mo K_{α} radiation ($\lambda = 0.71073 \text{ \AA}$) at 293 K. Intensity data for 440 reflections ($h = -17$ to 11, $k = -15$ to 17, $l = -13$ to 13) were collected in the 2θ scan mode in the range $5.12 < 2\theta < 46.46$. From 440 independent reflections ($R_{\text{int}} = 0.0568$), 388 were observed with $I > 2\sigma(I)$. Intensity data were corrected for Lorentz and polarisation, but not for absorption. The crystal corresponds to the space group R-3c. The structure was solved by direct methods using Siemens SHELXTL PLUS (PC version 5.1) programs [11]. Positions for the hydrogen atoms were calculated geometrically using the riding model with fixed isotropic temperature factors. Refinement of F^2 was done against all reflections. The weighted R factor wR and goodness of fit S are based on F^2 . Values of the final indices obtained were $R = 0.0568$ and $wR = 0.0604$ with $S = 1.1600$. A final difference Fourier electron-density map showed maximum and minimum peaks of 0.29 and 2733.0 e\AA^{-3} , respectively. Atomic scattering factors, dispersion corrections, and absorption coefficients were taken from the *International Tables for Crystallography* (1992, Vol. C).

Results and discussion

The inclusion compound Q(3 thiourea) is easily separated from thiourea because the hexagonal long needles of the inclusion compound crystallise before the tetragonal prisms of thiourea from methanol solutions. Furthermore, crystals of the inclusion compound have characteristic physical shapes very different from those of pure thiourea, and they may be easily distinguished and mechanically separated (Pasteur's method).

The structure of the studied compound was determined. As shown in Figure 1, the product may be described as a series of linear, infinite, non-intersecting hexagonal channels in which the quinuclidine guest molecules are located.

Table 1. Structural parameters and selected bond distances and angles to the matrix of the inclusion of (Q) in thiourea

	Q(3 thiourea)
Crystal system	Hexagonal
Space group	R-3c
Empirical formula	$\text{C}_{10}\text{H}_{25}\text{N}_7\text{S}_3$
$a \text{ \AA}$	15.94(2)
$b \text{ \AA}$	15.94(2)
$c \text{ \AA}$	12.43(2)
α°	90(2)
β°	90(2)
γ°	120(2)
Cell volume (\AA^3)	2733.0 (8)
Z (formula units/cell)	1
Density (calculated) (g/ml)	1.183
Absorption coefficient ($\text{m}\mu, \text{mm}^{-1}$)	0.365
$F(000)$	1028
Radiation source	Mo K_{α}
Wavelength \AA	0.71073
Data collection temperature ($^\circ\text{K}$)	293(2)
Two-theta range ($^\circ$)	5.12–46.46
Index ranges	$-17 < h < 11$ $-15 < k < 17$ $-13 < l < 13$
Reflection collected	440
Observed reflection	388
Residuals (observed data)	0.0568
Residuals (all data)	0.0604
Bond lengths (\AA)	
S—C	1.726(4)
C—N	1.308(3)
Bond angles ($^\circ$)	
N—C—N	119.7(4)
N—C—S	120.1(2)

However, due to substantial disorder of the quinuclidine molecule, neither its geometry nor orientation could be exactly determined. Nonetheless, the basic structure appears to be commensurate.

Structural parameters for the matrix as well as most relevant bond distances and angles are given in Table 1.

In comparison with other inclusion compounds, the topotactic nature of the corresponding formation reaction (host + guest = inclusion compound), is rather limited in the case of most clathrates; the existence of the matrix structure usually needs the presence of the guest, so the escape of the guest leads to the destruction of the host structure. However, in the quinuclidine-thiourea clathrate discussed here, as shown by its thermal behaviour, topotactic properties of the system appear to be present to some extent. Indeed, the differential thermal analysis of the product illustrated in Figure 2 shows a single relatively wide, slightly asymmetric strong peak centered at 175.6°C , the melting temperature of thiourea [12]. This behaviour is rather different from that observed for the cyclohexane-thiourea compound in which, in addition to the melting of the thiourea, an asymmetric peak at $140\text{--}160^\circ\text{C}$ due to the disintegration of the complex is observed [13]. The asymmetry of the peak observed

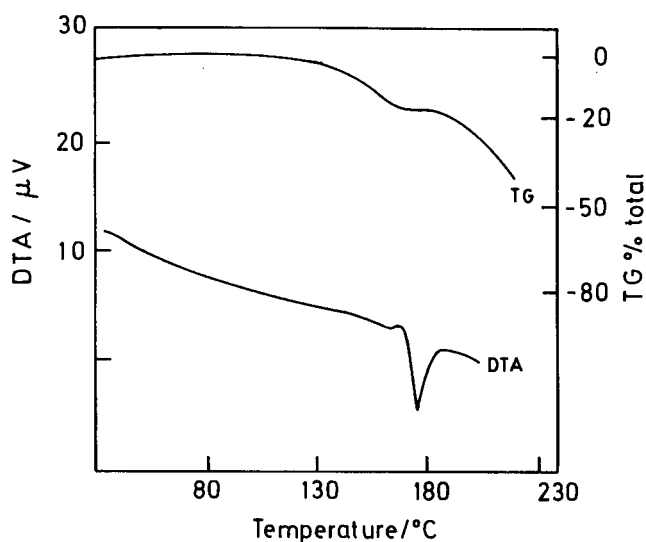


Figure 2. Thermal analysis, DTA and TG of Q(3 thiourea).

in the DTA (Figure 2) may be attributed to an additional process, also endothermic but with a relatively lower heat tone, occurring between 160 and 170 °C, which could be assigned to the decomposition of the clathrate to orthorhombic thiourea. The interpretation of this feature as a thermal process in which the compound is stable enough for enduring with the same structure until near the melting point of the thiourea is confirmed by the TG in which the partial loss of the guest begins at 115–130 °C. At 160 °C, when 40% of the originally inserted amine is lost, a clear change of the mass loss rate is observed. About 60% of the amine remains retained by the thiourea melt after the decomposition of the solid. These experiments suggest that the thiourea host structure does not experience decomposition before melting, and those thiourea-amine inclusion compounds possess an open host structure.

The long needle crystals undergo an interesting anisotropic dissolution process in contact with a high polarity solvent such as DMSO or water [10]: the dissolving of the matrix first by the needle ends of the crystals, and the corresponding escape of the guest propels the crystals through the liquid surface. The evolution of the amine may be observed visually by adding phenolphthalein to the solution. The motion effect is thus accompanied by the formation of a beautiful red “wake” in the liquid.

For ^{13}C nuclei bonded directly to nitrogen (^{14}N , $I = 1$), MAS frequently gives NMR patterns consisting of approximately 1:2 (or 2:1) doublets because ^{13}C - ^{14}N residual dipolar interactions are averaged to zero by ^{13}C MAS NMR. In the nitrogen compounds the axis of quantisation of the ^{14}N nucleus is tilted from the direction of the magnetic field as a consequence of the interaction between the ^{14}N nuclear quadrupole moment and the electric field gradient at the same nucleus [7, 14–17].

As shown in Figure 3, which reproduces the ^{13}C CP-MAS NMR spectrum of the studied inclusion compound at room temperature, high amplitude conformational, rotational and probably translational motions of the guest molecules in the channel average the residual dipolar in-

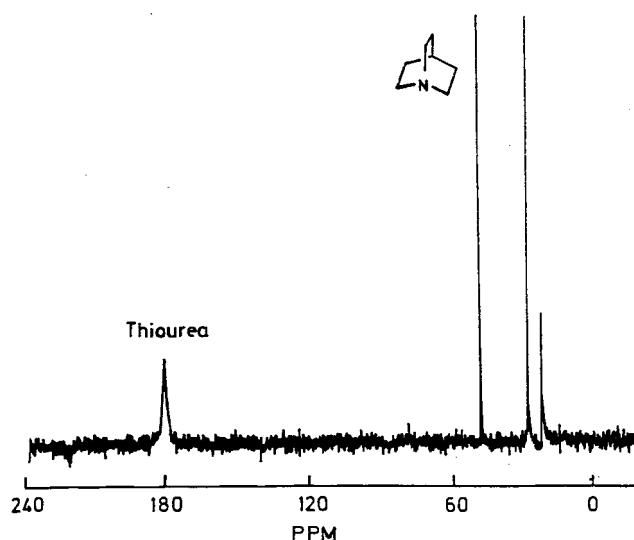


Figure 3. ^{13}C CP-MAS NMR spectrum of the Q(3 thiourea).

Table 2. Medium effects on ^{13}C -NMR chemical shifts in quinuclidine

C-atom in Quinuclidine	^{13}C -NMR chemical shifts (ppm) in			
	CCl_4 (10% p/v)	CDCl_3 (1% p/v)	CDCl_3 (10% p/v)	Thiourea
C-1	47.92	47.83	47.79	48.27
C-2	27.37	26.73	26.76	27.80
C-3	21.33	20.76	20.88	21.74

teractions between ^{13}C and ^{14}N to less than 10 Hz, the spectrum showing only a single sharp line for C, so the typical asymmetric doublet is not observed.

The classic clathrate structure as well as the topotactic nature of the compound discussed above implies a relatively weak amine-thiourea host interaction which does not compete with the H-bond interactions involved in the existence of the thiourea matrix. The relatively high movement of the guest observed in the X-ray diffraction pattern commented above may also be seen in the ^{13}C NMR spectrum of the solid.

Table 2 compares the chemical shifts of quinuclidine inserted in the thiourea matrix with those of the amine in other media. The behaviour of organic molecules placed in environments like those within clathrate cavities is expected, in general, to differ from that of the same molecules in other phases [18–20]. In previous work, the ^{13}C NMR behaviour of quinuclidine has been analysed in detail [8]. Indeed, the effect of the amine self-association on the chemical shifts can be seen by observing the shift on going from the quinuclidine crystals to the dilute solution in CDCl_3 . Interestingly, the ^{13}C -chemical shifts of quinuclidine inserted in thiourea correspond to the free amine or, at least, to an amine undergoing weaker interactions with the medium than the amine dissolved in an inert solvent. Thiourea channels appear to be involved in this ternary bicyclic amine, a perfect van der Waals cavity.

Supplementary tables

Table 1a. Atomic coordinates ($\text{Å} \times 10^4$) and equivalent isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (eq)
S	6667	6342(1)	833	51(1)
C	6667	7424(2)	833	49(1)
N	6471(2)	7738(2)	1718(2)	67(1)
HA	6410(20)	7460(20)	2320(30)	70(10)
HB	6540(30)	8300(20)	1760(30)	80(10)
C(1)	9280(20)	9710(30)	1440(30)	72(3)
C(2)	8960(20)	9360(20)	3090(20)	72(3)
C(3)	9010(20)	9600(20)	2490(30)	72(3)
C(4)	9090(20)	9090(20)	2500	72(3)
C(5)	9070(20)	9240(30)	1820(30)	72(3)
C(6)	10000	10000	1440(30)	72(3)
C(7)	9070(20)	9830(20)	1880(40)	72(3)
C(8)	9700(30)	10480(30)	1440(40)	72(3)

U (eq) is defined as 1/3 the trace of the U_{ij} tensor.

Table 1b. Bond lengths (Å) and angles (deg) for

S—C	1.723(4)	C—N#18	1.308(3)
C—N	1.308(3)	C(1)—C(7)	0.71(4)
C(1)—C(5)	0.80(4)	C(1)—C(6)	1.00(3)
C(1)—C(8)#2	1.03(4)	C(1)—C(8)	1.07(5)
C(1)—C(3)	1.36(5)	C(1)—C(2)#4	1.39(4)
C(1)—C(2)#5	1.43(5)	C(1)—C(4)	1.58(4)
C(1)—C(3)#5	1.67(5)	C(1)—C(1)#3	1.74(6)
C(1)—C(1)#2	1.74(6)	C(2)—C(8)#5	0.68(4)
C(2)—C(5)#4	0.79(3)	C(2)—C(3)	0.81(3)
C(2)—C(4)	0.92(2)	C(2)—C(7)#5	1.14(3)
C(2)—C(1)#4	1.39(4)	C(2)—C(1)#5	1.43(5)
C(2)—C(6)#4	1.56(3)	C(2)—C(3)#4	1.55(3)
C(2)—C(3)#5	1.62(5)	C(2)—C(5)	1.61(4)
C(2)—C(7)#4	1.65(4)	C(3)—C(7)	0.83(4)
C(3)—C(4)	0.88(2)	C(3)—C(5)	1.05(4)
C(3)—C(3)#5	1.12(4)	C(3)—C(7)#5	1.11(5)
C(3)—C(5)#4	1.36(5)	C(3)—C(8)#5	1.38(6)
C(3)—C(2)#4	1.55(3)	C(3)—C(3)#4	1.61(5)
C(3)—C(2)#5	1.62(5)	C(4)—C(3)#4	0.88(2)
C(4)—C(5)#4	0.89(3)	C(4)—C(5)	0.89(3)
C(4)—C(2)#4	0.92(2)	C(4)—C(7)#4	1.41(4)
C(4)—C(7)	1.42(4)	C(4)—C(8)#5	1.45(5)
C(4)—C(8)#2	1.45(5)	C(4)—C(1)#4	1.58(4)
C(5)—C(2)#4	0.79(3)	C(5)—C(8)#2	0.86(4)
C(5)—C(7)	0.94(4)	C(5)—C(3)#4	1.36(5)
C(5)—C(6)	1.44(3)	C(5)—C(7)#2	1.75(4)
C(5)—C(8)	1.78(5)	C(5)—C(5)#4	1.76(7)
C(6)—C(1)#3	1.00(3)	C(6)—C(1)#2	1.00(3)
C(6)—C(8)#2	1.09(4)	C(6)—C(8)#3	1.09(4)
C(6)—C(8)	1.09(4)	C(6)—C(5)#3	1.44(3)
C(6)—C(5)#2	1.44(3)	C(6)—C(7)#3	1.47(4)
C(6)—C(7)#2	1.47(4)	C(6)—C(7)	1.47(4)
C(7)—C(3)#5	1.11(5)	C(7)—C(2)#5	1.14(3)
C(7)—C(8)	1.16(6)	C(7)—C(8)#2	1.56(5)

Table 1b. Continued

C(7)—C(7)#5	1.6(1)	C(7)—C(2)#4	1.65(4)
C(8)—C(2)#5	0.68(4)	C(8)—C(5)#3	0.86(4)
C(8)—C(1)#3	1.03(4)	C(8)—C(3)#5	1.38(6)
C(8)—C(4)#3	1.45(5)	C(8)—C(7)#3	1.56(5)
C(8)—C(3)#3	1.79(5)		
N—C#18—N	119.7(4)	N—C#18—S	120.1(2)
N—C—S	120.1(2)	C(7)—C(1)—C(5)	77(5)
C(7)—C(1)—C(6)	116(5)	C(5)—C(1)—C(6)	106(4)
C(7)—C(1)—C(8)#2	126(6)	C(5)—C(1)—C(8)#2	55(4)
C(6)—C(1)—C(8)#2	65(3)	C(7)—C(1)—C(8)	78(6)
C(5)—C(1)—C(8)	143(6)	C(6)—C(1)—C(8)	63(3)
C(8)—C(1)#2—C(8)	128(5)	C(7)—C(1)—C(3)	30(4)
C(5)—C(1)—C(3)	50(3)	C(6)—C(1)—C(3)	106(4)
C(8)—C(1)#2—C(3)	96(4)	C(8)—C(1)—C(3)	97(5)
C(7)—C(1)—C(2)#4	99(5)	C(5)—C(1)—C(2)#4	29(3)
C(6)—C(1)—C(2)#4	80(2)	C(8)—C(1)#2—C(2)#4	28(3)
C(8)—C(1)—C(2)#4	135(3)	C(3)—C(1)—C(2)#4	69(2)
C(7)—C(1)—C(2)#5	52(4)	C(5)—C(1)—C(2)#5	120(5)
C(6)—C(1)—C(2)#5	77(2)	C(8)—C(1)#2—C(2)#5	135(3)
C(8)—C(1)—C(2)#5	27(3)	C(3)—C(1)—C(2)#5	71(3)
C(2)—C(1)#4—C(2)#5	125(3)	C(7)—C(1)—C(4)	64(4)
C(5)—C(1)—C(4)	21(3)	C(6)—C(1)—C(4)	96(3)
C(8)—C(1)#2—C(4)	63(4)	C(8)—C(1)—C(4)	122(4)
C(3)—C(1)—C(4)	34(1)	C(2)—C(1)#4—C(4)	35(1)
C(2)—C(1)#5—C(4)	100(3)	C(7)—C(1)—C(3)#5	30(3)
C(5)—C(1)—C(3)#5	91(4)	C(6)—C(1)—C(3)#5	87(3)
C(8)—C(1)#2—C(3)#5	122(4)	C(8)—C(1)—C(3)#5	55(4)
C(3)—C(1)—C(3)#5	42(2)	C(2)—C(1)#4—C(3)#5	101(3)
C(2)—C(1)#5—C(3)#5	29(1)	C(4)—C(1)—C(3)#5	71(2)
C(7)—C(1)—C(1)#3	99(5)	C(5)—C(1)—C(1)#3	128(3)
C(6)—C(1)—C(1)#3	30.00(1)	C(8)—C(1)#2—C(1)#3	95(3)
C(8)—C(1)—C(1)#3	33(3)	C(3)—C(1)—C(1)#3	104(2)
C(2)—C(1)#4—C(1)#3	107(2)	C(2)—C(1)#5—C(1)#3	51(2)
C(4)—C(1)—C(1)#3	111(2)	C(3)—C(1)#5—C(1)#3	70(2)
C(7)—C(1)—C(1)#2	128(4)	C(5)—C(1)—C(1)#2	81(4)
C(6)—C(1)—C(1)#2	30.00(1)	C(8)—C(1)#2—C(1)#2	35(3)
C(8)—C(1)—C(1)#2	93(3)	C(3)—C(1)—C(1)#2	103(2)
C(2)—C(1)#4—C(1)#2	53(3)	C(2)—C(1)#5—C(1)#2	105(2)
C(4)—C(1)—C(1)#2	80(2)	C(3)—C(1)#5—C(1)#2	105(2)
C(1)—C(1)#3—C(1)#2	60.00(2)	C(8)—C(2)#5—C(5)#4	71(4)
C(8)—C(2)#5—C(3)	135(5)	C(5)—C(2)#4—C(3)	117(4)
C(8)—C(2)#5—C(4)	128(5)	C(5)—C(2)#4—C(4)	62(3)
C(3)—C(2)—C(4)	61(2)	C(8)—C(2)#5—C(7)#5	74(5)
C(5)—C(2)#4—C(7)#5	129(4)	C(3)—C(2)—C(7)#5	67(3)
C(4)—C(2)—C(7)#5	120(4)	C(8)—C(2)#5—C(1)#4	45(4)
C(5)—C(2)#4—C(1)#4	29(3)	C(3)—C(2)—C(1)#4	123(3)
C(4)—C(2)—C(1)#4	84(3)	C(7)—C(2)#5—C(1)#4	103(4)
C(8)—C(2)#5—C(1)#5	46(4)	C(5)—C(2)#4—C(1)#5	105(4)
C(3)—C(2)—C(1)#5	92(3)	C(4)—C(2)—C(1)#5	129(3)
C(7)—C(2)#5—C(1)#5	29(2)	C(1)—C(2)#4—C(1)#5	76(3)
C(8)—C(2)#5—C(6)#4	37(3)	C(5)—C(2)#4—C(6)#4	67(3)
C(3)—C(2)—C(6)#4	102(3)	C(4)—C(2)—C(6)#4	102(3)
C(7)—C(2)#5—C(6)#4	64(2)	C(1)—C(2)#4—C(6)#4	39(2)
C(1)—C(2)#5—C(6)#4	39(2)	C(8)—C(2)#5—C(3)#4	99(4)
C(5)—C(2)#4—C(3)#4	38(2)	C(3)—C(2)—C(3)#4	79(3)
C(4)—C(2)—C(3)#4	30(2)	C(7)—C(2)#5—C(3)#4	117(3)
C(1)—C(2)#4—C(3)#4	55(2)	C(1)—C(2)#5—C(3)#4	110(2)

Table 1b. Continued

C(6)—C(2)#4—C(3)#4	75(2)	C(8)—C(2)#5—C(3)#5	97(4)
C(5)—C(2)#4—C(3)#5	125(3)	C(3)—C(2)—C(3)#5	39(3)
C(4)—C(2)—C(3)#5	92(3)	C(7)—C(2)#5—C(3)#5	29(2)
C(1)—C(2)#4—C(3)#5	109(2)	C(1)—C(2)#5—C(3)#5	53(2)
C(6)—C(2)#4—C(3)#5	73(1)	C(3)—C(2)#4—C(3)#5	97(3)
C(8)—C(2)#5—C(5)	143(4)	C(5)—C(2)#4—C(5)	87(3)
C(3)—C(2)—C(5)	34(2)	C(4)—C(2)—C(5)	27(2)
C(7)—C(2)#5—C(5)	99(4)	C(1)—C(2)#4—C(5)	105(3)
C(1)—C(2)#5—C(5)	118(2)	C(6)—C(2)#4—C(5)	108(2)
C(3)—C(2)#4—C(5)	51(2)	C(3)—C(2)#5—C(5)	70(3)
C(8)—C(2)#5—C(7)#4	70(4)	C(5)—C(2)#4—C(7)#4	18(2)
C(3)—C(2)—C(7)#4	104(3)	C(4)—C(2)—C(7)#4	59(2)
C(7)—C(2)#5—C(7)#	114(3)	C(1)—C(2)#4—C(7)#4	25(2)
C(1)—C(2)#5—C(7)#4	93(3)	C(6)—C(2)#4—C(7)#4	54(2)
C(3)—C(2)#4—C(7)#4	30(2)	C(3)—C(2)#5—C(7)#4	107(2)
C(5)—C(2)—C(7)#4	80(3)	C(2)—C(3)—C(7)	178(5)
C(2)—C(3)—C(4)	66(3)	C(7)—C(3)—C(4)	112(4)
C(2)—C(3)—C(5)	119(3)	C(7)—C(3)—C(5)	59(4)
C(4)—C(3)—C(5)	54(2)	C(2)—C(3)—C(3)#5	113(5)
C(7)—C(3)—C(3)#5	68(5)	C(4)—C(3)—C(3)#5	143(3)
C(5)—C(3)—C(3)#5	119(5)	C(2)—C(3)—C(7)#5	71(4)
C(7)—C(3)—C(7)#5	111(5)	C(4)—C(3)—C(7)#5	128(5)
C(5)—C(3)—C(7)#5	157(4)	C(3)—C(3)#5—C(7)#5	44(2)
C(2)—C(3)—C(5)#4	31(2)	C(7)—C(3)—C(5)#4	147(4)
C(4)—C(3)—C(5)#4	40(2)	C(5)—C(3)—C(5)#4	93(3)
C(3)—C(3)#5—C(5)#4	121(3)	C(7)—C(3)#5—C(5)#4	89(4)
C(2)—C(3)—C(1)	152(3)	C(7)—C(3)—C(1)	26(3)
C(4)—C(3)—C(1)	87(3)	C(5)—C(3)—C(1)	36(2)
C(3)—C(3)#5—C(1)	84(4)	C(7)—C(3)#5—C(1)	127(4)
C(5)—C(3)#4—C(1)	121(3)	C(2)—C(3)—C(8)#5	21(2)
C(7)—C(3)—C(8)#5	159(4)	C(4)—C(3)—C(8)#5	76(3)
C(5)—C(3)—C(8)#5	129(3)	C(3)—C(3)#5—C(8)#5	94(4)
C(7)—C(3)#5—C(8)#5	54(3)	C(5)—C(3)#4—C(8)#5	37(2)
C(1)—C(3)—C(8)#5	149(3)	C(2)—C(3)—C(2)#4	96(3)
C(7)—C(3)—C(2)#4	82(4)	C(4)—C(3)—C(2)#4	31(2)
C(5)—C(3)—C(2)#4	27(2)	C(3)—C(3)#5—C(2)#4	125(3)
C(7)—C(3)#5—C(2)#4	141(3)	C(5)—C(3)#4—C(2)#4	67(2)
C(1)—C(3)—C(2)#4	56(2)	C(8)—C(3)#5—C(2)#4	102(3)
C(2)—C(3)—C(3)#4	71(3)	C(7)—C(3)—C(3)#4	107(4)
C(4)—C(3)—C(3)#4	23(3)	C(5)—C(3)—C(3)#4	57(2)
C(3)—C(3)#5—C(3)#4	120.0(1)	C(7)—C(3)#5—C(3)#4	115(4)
C(5)—C(3)#4—C(3)#4	40(2)	C(1)—C(3)—C(3)#4	81(3)
C(8)—C(3)#5—C(3)#4	73(3)	C(2)—C(3)#4—C(3)#4	30(1)
C(2)—C(3)—C(2)#5	139(3)	C(7)—C(3)—C(2)#5	42(3)
C(4)—C(3)—C(2)#5	133(3)	C(5)—C(3)—C(2)#5	92(3)
C(3)—C(3)#5—C(2)#5	28(2)	C(7)—C(3)#5—C(2)#5	71(3)
C(5)—C(3)#4—C(2)#5	135(2)	C(1)—C(3)—C(2)#5	57(2)
C(8)—C(3)#5—C(2)#5	119(2)	C(2)—C(3)#4—C(2)#5	104(3)
C(3)—C(3)#4—C(2)#5	113(2)	C(3)—C(4)#4—C(3)	133(5)
C(3)—C(4)#4—C(5)#4	73(3)	C(3)—C(4)—C(5)#4	101(3)
C(3)—C(4)#4—C(5)	101(3)	C(3)—C(4)—C(5)	73(3)
C(5)—C(4)#4—C(5)	165(6)	C(3)—C(4)#4—C(2)	119(3)
C(3)—C(4)—C(2)	54(2)	C(5)—C(4)#4—C(2)	52(2)
C(5)—C(4)—C(2)	126(2)	C(3)—C(4)#4—C(2)#4	54(2)
C(3)—C(4)—C(2)#4	119(3)	C(5)—C(4)#4—C(2)#4	126(3)
C(5)—C(4)—C(2)#4	52(2)	C(2)—C(4)—C(2)#4	165(5)
C(3)—C(4)#4—C(7)#4	33(3)	C(3)—C(4)—C(7)#4	121(3)
C(5)—C(4)#4—C(7)#4	41(3)	C(5)—C(4)—C(7)#4	131(3)

Table 1b. Continued

C(2)—C(4)—C(7)#4	87(3)	C(2)—C(4)#4—C(7)#4	87(3)
C(3)—C(4)#4—C(7)	121(3)	C(3)—C(4)—C(7)	33(3)
C(5)—C(4)#4—C(7)	131(3)	C(5)—C(4)—C(7)	40(3)
C(2)—C(4)—C(7)	86(3)	C(2)—C(4)#4—C(7)	87(3)
C(7)—C(4)#4—C(7)	131(3)	C(3)—C(4)#4—C(8)#5	98(3)
C(3)—C(4)—C(8)#5	68(3)	C(5)—C(4)#4—C(8)#5	33(2)
C(5)—C(4)—C(8)#5	139(3)	C(2)—C(4)—C(8)#5	22(2)
C(2)—C(4)#4—C(8)#5	148(3)	C(7)—C(4)#4—C(8)#5	66(2)
C(7)—C(4)—C(8)#5	99(3)	C(3)—C(4)#4—C(8)#2	68(3)
C(3)—C(4)—C(8)#2	98(3)	C(5)—C(4)#4—C(8)#2	139(3)
C(5)—C(4)—C(8)#2	34(2)	C(2)—C(4)—C(8)#2	148(3)
C(2)—C(4)#4—C(8)#2	22(2)	C(7)—C(4)#4—C(8)#2	99(3)
C(7)—C(4)—C(8)#2	66(2)	C(8)—C(4)#5—C(8)#2	144(4)
C(3)—C(4)#4—C(1)#4	59(3)	C(3)—C(4)—C(1)#4	101(3)
C(5)—C(4)#4—C(1)#4	19(3)	C(5)—C(4)—C(1)#4	147(4)
C(2)—C(4)—C(1)#4	61(2)	C(2)—C(4)#4—C(1)#4	113(3)
C(7)—C(4)#4—C(1)#4	27(2)	C(7)—C(4)—C(1)#4	124(2)
C(8)—C(4)#5—C(1)#4	39(2)	C(8)—C(4)#2—C(1)#4	121(2)
C(3)—C(4)#4—C(1)	101(3)	C(3)—C(4)—C(1)	59(3)
C(5)—C(4)#4—C(1)	147(4)	C(5)—C(4)—C(1)	19(3)
C(2)—C(4)—C(1)	112(3)	C(2)—C(4)#4—C(1)	61(2)
C(7)—C(4)#4—C(1)	124(2)	C(7)—C(4)—C(1)	27(2)
C(8)—C(4)#5—C(1)	121(2)	C(8)—C(4)#2—C(1)	39(2)
C(1)—C(4)#4—C(1)	132(3)	C(2)—C(5)#4—C(1)	122(5)
C(2)—C(5)#4—C(8)#2	49(3)	C(1)—C(5)—C(8)#2	76(5)
C(2)—C(5)#4—C(4)	66(3)	C(1)—C(5)—C(4)	140(5)
C(8)—C(5)#2—C(4)	112(5)	C(2)—C(5)#4—C(7)	147(4)
C(1)—C(5)—C(7)	47(3)	C(8)—C(5)#2—C(7)	120(5)
C(4)—C(5)—C(7)	102(4)	C(2)—C(5)#4—C(3)	115(4)
C(1)—C(5)—C(3)	94(5)	C(8)—C(5)#2—C(3)	139(4)
C(4)—C(5)—C(3)	53(2)	C(7)—C(5)—C(3)	49(3)
C(2)—C(5)#4—C(3)#4	32(2)	C(1)—C(5)—C(3)#4	127(4)
C(8)—C(5)#2—C(3)#4	73(4)	C(4)—C(5)—C(3)#4	39(2)
C(7)—C(5)—C(3)#4	121(3)	C(3)—C(5)—C(3)#4	83(2)
C(2)—C(5)#4—C(6)	83(3)	C(1)—C(5)—C(6)	42(3)
C(8)—C(5)#2—C(6)	49(3)	C(4)—C(5)—C(6)	113(3)
C(7)—C(5)—C(6)	73(3)	C(3)—C(5)—C(6)	98(3)
C(3)—C(5)#4—C(6)	86(2)	C(2)—C(5)#4—C(2)	93(3)
C(1)—C(5)—C(2)	119(5)	C(8)—C(5)#2—C(2)	132(4)
C(4)—C(5)—C(2)	28(1)	C(7)—C(5)—C(2)	75(4)
C(3)—C(5)—C(2)	26(2)	C(3)—C(5)#4—C(2)	62(2)
C(6)—C(5)—C(2)	110(2)	C(2)—C(5)#4—C(7)#2	30(3)
C(1)—C(5)—C(7)#2	95(4)	C(8)—C(5)#2—C(7)#2	35(4)
C(4)—C(5)—C(7)#2	78(3)	C(7)—C(5)—C(7)#2	119(3)
C(3)—C(5)—C(7)#2	109(3)	C(3)—C(5)#4—C(7)#2	40(2)
C(6)—C(5)—C(7)#2	54(2)	C(2)—C(5)—C(7)#2	97(2)
C(2)—C(5)#4—C(8)	120(4)	C(1)—C(5)—C(8)	21(3)
C(8)—C(5)#2—C(8)	84(5)	C(4)—C(5)—C(8)	119(4)
C(7)—C(5)—C(8)	36(3)	C(3)—C(5)—C(8)	76(3)
C(3)—C(5)#4—C(8)	113(2)	C(6)—C(5)—C(8)	38(2)
C(2)—C(5)—C(8)	99(3)	C(7)—C(5)#2—C(8)	90(2)
C(2)—C(5)#4—C(5)#4	66(3)	C(1)—C(5)—C(5)#4	133(4)
C(8)—C(5)#2—C(5)#4	109(5)	C(4)—C(5)—C(5)#4	7(3)
C(7)—C(5)—C(5)#4	98(4)	C(3)—C(5)—C(5)#4	51(2)
C(3)—C(5)#4—C(5)#4	37(2)	C(6)—C(5)—C(5)#4	106(2)
C(2)—C(5)—C(5)#4	27(1)	C(7)—C(5)#2—C(5)#4	74(3)
C(8)—C(5)—C(5)#4	112(2)	C(1)—C(6)#3—C(1)	120.00(3)
C(1)—C(6)#3—C(1)#2	120.00(5)	C(1)—C(6)—C(1)#2	120.00(3)

Table 1b. Continued

C(1)—C(6)#3—C(8)#2	178(2)	C(1)—C(6)—C(8)#2	58(2)
C(1)—C(6)#2—C(8)#2	62(2)	C(1)—C(6)#3—C(8)#3	62(2)
C(1)—C(6)—C(8)#3	178(2)	C(1)—C(6)#2—C(8)#3	58(2)
C(8)—C(6)#2—C(8)#3	120.00(5)	C(1)—C(6)#3—C(8)	58(2)
C(1)—C(6)—C(8)	62(2)	C(1)—C(6)#2—C(8)	178(3)
C(8)—C(6)#2—C(8)	120.00(3)	C(8)—C(6)#3—C(8)	120.00(6)
C(1)—C(6)#3—C(5)#3	32(2)	C(1)—C(6)—C(5)#3	93(3)
C(1)—C(6)#2—C(5)#3	142(3)	C(8)—C(6)#2—C(5)#3	147(3)
C(8)—C(6)#3—C(5)#3	88(2)	C(8)—C(6)—C(5)#3	37(2)
C(1)—C(6)#3—C(5)#2	93(3)	C(1)—C(6)—C(5)#2	142(3)
C(1)—C(6)#2—C(5)#2	32(2)	C(8)—C(6)#2—C(5)#2	88(2)
C(8)—C(6)#3—C(5)#2	37(2)	C(8)—C(6)—C(5)#2	147(3)
C(5)—C(6)#3—C(5)#2	110(2)	C(1)—C(6)#3—C(5)	142(3)
C(1)—C(6)—C(5)	32(2)	C(1)—C(6)#2—C(5)	93(3)
C(8)—C(6)#2—C(5)	37(2)	C(8)—C(6)#3—C(5)	147(3)
C(8)—C(6)—C(5)	88(2)	C(5)—C(6)#3—C(5)	110(2)
C(5)—C(6)#2—C(5)	110(2)	C(1)—C(6)#3—C(7)#3	26(2)
C(1)—C(6)—C(7)#3	130(3)	C(1)—C(6)#2—C(7)#3	105(3)
C(8)—C(6)#2—C(7)#3	155(4)	C(8)—C(6)#3—C(7)#3	51(3)
C(8)—C(6)—C(7)#3	73(3)	C(5)—C(6)#3—C(7)#3	38(2)
C(5)—C(6)#2—C(7)#3	74(2)	C(5)—C(6)—C(7)#3	135(3)
C(1)—C(6)#3—C(7)#2	130(3)	C(1)—C(6)—C(7)#2	105(3)
C(1)—C(6)#2—C(7)#2	26(2)	C(8)—C(6)#2—C(7)#2	51(3)
C(8)—C(6)#3—C(7)#2	73(3)	C(8)—C(6)—C(7)#2	155(4)
C(5)—C(6)#3—C(7)#2	135(3)	C(5)—C(6)#2—C(7)#2	38(2)
C(5)—C(6)—C(7)#2	74(2)	C(7)—C(6)#3—C(7)#2	107(2)
C(1)—C(6)#3—C(7)	105(3)	C(1)—C(6)—C(7)	26(2)
C(1)—C(6)#2—C(7)	130(3)	C(8)—C(6)#2—C(7)	73(3)
C(8)—C(6)#3—C(7)	155(4)	C(8)—C(6)—C(7)	51(3)
C(5)—C(6)#3—C(7)	74(2)	C(5)—C(6)#2—C(7)	135(3)
C(5)—C(6)—C(7)	38(2)	C(7)—C(6)#3—C(7)	107(2)
C(7)—C(6)#2—C(7)	107(2)	C(1)—C(7)—C(3)	124(6)
C(1)—C(7)—C(5)	56(4)	C(3)—C(7)—C(5)	72(4)
C(1)—C(7)—C(3)#5	131(5)	C(3)—C(7)—C(3)#5	68(5)
C(5)—C(7)—C(3)#5	130(4)	C(1)—C(7)—C(2)#5	99(5)
C(3)—C(7)—C(2)#5	110(5)	C(5)—C(7)—C(2)#5	140(4)
C(3)—C(7)#5—C(2)#5	42(2)	C(1)—C(7)—C(8)	65(5)
C(3)—C(7)—C(8)	134(4)	C(5)—C(7)—C(8)	115(5)
C(3)—C(7)#5—C(8)	75(4)	C(2)—C(7)#5—C(8)	34(2)
C(1)—C(7)—C(4)	90(5)	C(3)—C(7)—C(4)	35(2)
C(5)—C(7)—C(4)	38(2)	C(3)—C(7)#5—C(4)	96(4)
C(2)—C(7)#5—C(4)	128(3)	C(8)—C(7)—C(4)	130(3)
C(1)—C(7)—C(6)	38(3)	C(3)—C(7)—C(6)	108(3)
C(5)—C(7)—C(6)	70(3)	C(3)—C(7)#5—C(6)	94(2)
C(2)—C(7)#5—C(6)	72(2)	C(8)—C(7)—C(6)	47(3)
C(4)—C(7)—C(6)	86(2)	C(1)—C(7)—C(8)#2	32(4)
C(3)—C(7)—C(8)#2	92(4)	C(5)—C(7)—C(8)#2	29(3)
C(3)—C(7)#5—C(8)#2	125(3)	C(2)—C(7)#5—C(8)#2	114(4)
C(8)—C(7)—C(8)#2	87(5)	C(4)—C(7)—C(8)#2	58(2)
C(6)—C(7)—C(8)#2	42(2)	C(1)—C(7)—C(7)#5	144(4)
C(3)—C(7)—C(7)#5	40(3)	C(5)—C(7)—C(7)#5	110(4)
C(3)—C(7)#5—C(7)#5	29(2)	C(2)—C(7)#5—C(7)#5	71(3)
C(8)—C(7)—C(7)#5	103(4)	C(4)—C(7)—C(7)#5	73(3)
C(6)—C(7)—C(7)#5	108(2)	C(8)—C(7)#2—C(7)#5	120(3)
C(1)—C(7)—C(2)#4	56(4)	C(3)—C(7)—C(2)#4	68(3)
C(5)—C(7)—C(2)#4	15(2)	C(3)—C(7)#5—C(2)#4	117(3)
C(2)—C(7)#5—C(2)#4	126(3)	C(8)—C(7)—C(2)#4	107(4)
C(4)—C(7)—C(2)#4	34(1)	C(6)—C(7)—C(2)#4	60(2)

Table 1b. Continued

C(8)—C(7)#2—C(2)#4	24(2)	C(7)—C(7)#5—C(2)#4	101(3)
C(1)—C(7)—C(2)	123(5)	C(3)—C(7)—C(2)	1(2)
C(5)—C(7)—C(2)	71(3)	C(3)—C(7)#5—C(2)	69(4)
C(2)—C(7)#5—C(2)	110(4)	C(8)—C(7)—C(2)	133(3)
C(4)—C(7)—C(2)	34(1)	C(6)—C(7)—C(2)	107(3)
C(8)—C(7)#2—C(2)	91(3)	C(7)—C(7)#5—C(2)	41(2)
C(2)—C(7)#4—C(2)	67(2)	C(2)—C(8)#5—C(5)#3	60(4)
C(2)—C(8)#5—C(1)#3	107(6)	C(5)—C(8)#3—C(1)#3	49(3)
C(2)—C(8)#5—C(1)	107(6)	C(5)—C(8)#3—C(1)	137(5)
C(1)—C(8)#3—C(1)	112(6)	C(2)—C(8)#5—C(6)	122(5)
C(5)—C(8)#3—C(6)	95(4)	C(1)—C(8)#3—C(6)	57(3)
C(1)—C(8)—C(6)	55(3)	C(2)—C(8)#5—C(7)	71(5)
C(5)—C(8)#3—C(7)	119(5)	C(1)—C(8)#3—C(7)	130(4)
C(1)—C(8)—C(7)	37(3)	C(6)—C(8)—C(7)	81(3)
C(2)—C(8)#5—C(3)#5	25(3)	C(5)—C(8)#3—C(3)#5	71(4)
C(1)—C(8)#3—C(3)#5	107(4)	C(1)—C(8)—C(3)#5	85(4)
C(6)—C(8)—C(3)#5	100(3)	C(7)—C(8)—C(3)#5	51(3)
C(2)—C(8)#5—C(4)#3	30(3)	C(5)—C(8)#3—C(4)#3	35(3)
C(1)—C(8)#3—C(4)#3	78(4)	C(1)—C(8)—C(4)#3	115(4)
C(6)—C(8)—C(4)#3	101(3)	C(7)—C(8)—C(4)#3	86(4)
C(3)—C(8)#5—C(4)#3	36(2)	C(2)—C(8)#5—C(7)#3	86(4)
C(5)—C(8)#3—C(7)#3	32(3)	C(1)—C(8)#3—C(7)#3	22(3)
C(1)—C(8)—C(7)#3	116(3)	C(6)—C(8)—C(7)#3	64(2)
C(7)—C(8)—C(7)#3	119(4)	C(3)—C(8)#5—C(7)#3	86(3)
C(4)—C(8)#3—C(7)#3	56(2)	C(2)—C(8)#5—C(5)	95(5)
C(5)—C(8)#3—C(5)	123(4)	C(1)—C(8)#3—C(5)	109(3)
C(1)—C(8)—C(5)	16(2)	C(6)—C(8)—C(5)	54(2)
C(7)—C(8)—C(5)	28(2)	C(3)—C(8)#5—C(5)	71(2)
C(4)—C(8)#3—C(5)	99(2)	C(7)—C(8)#3—C(5)	107(2)
C(2)—C(8)#5—C(3)#3	59(4)	C(5)—C(8)#3—C(3)#3	22(2)
C(1)—C(8)#3—C(3)#3	49(3)	C(1)—C(8)—C(3)#3	115(3)
C(6)—C(8)—C(3)#3	78(3)	C(7)—C(8)—C(3)#3	101(3)
C(3)—C(8)#5—C(3)#3	60(2)	C(4)—C(8)#3—C(3)#3	29(1)
C(7)—C(8)#3—C(3)#3	28(2)	C(5)—C(8)—C(3)#3	101(2)
C(2)—C(8)#5—C(3)	61(4)	C(5)—C(8)#3—C(3)	101(4)
C(1)—C(8)#3—C(3)	115(3)	C(1)—C(8)—C(3)	47(3)
C(6)—C(8)—C(3)	76(2)	C(7)—C(8)—C(3)	19(2)
C(3)—C(8)#5—C(3)	37(2)	C(4)—C(8)#3—C(3)	69(2)
C(7)—C(8)#3—C(3)	101(2)	C(5)—C(8)—C(3)	34(2)
C(3)—C(8)#3—C(3)	82(2)		

Estimated standard deviations are given in the parenthesis.

Symmetry operators:

1: x, y, z	2: $-y, x - y, z$
3: $-x + y, -x, z$	4: $y, x, -z + 1/2$
5: $x - y, -y, -z + 1/2$	6: $-x, -x + y, -z + 1/2$
7: $x + 2/3, y + 1/3, z + 1/3$	8: $-y + 2/3, x - y + 1/3, z + 1/3$
9: $-x + y + 2/3, -x + 1/3, z + 1/3$	10: $y + 2/3, x + 1/3, -z + 5/6$
11: $x - y + 2/3, -y + 1/3, -z + 5/6$	12: $-x + 2/3, -x + y + 1/3, -z + 5/6$
13: $x + 1/3, y + 2/3, z + 2/3$	14: $-y + 1/3, x - y + 2/3, z + 2/3$
15: $-x + y + 1/3, -x + 2/3, z + 2/3$	16: $y + 1/3, x + 2/3, -z + 7/6$
17: $x - y + 1/3, -y + 2/3, -z + 7/6$	18: $-x + 1/3, -x + y + 2/3, -z + 7/6$
19: $-x, -y, -z$	20: $y, -x + y, -z$
21: $x - y, x, -z$	22: $-y, -x, z - 1/2$
23: $-x + y, y, z - 1/2$	24: $x, x - y, z - 1/2$
25: $-x + 2/3, -y + 1/3, -z + 1/3$	26: $y + 2/3, -x + y + 1/3, -z + 1/3$
27: $x - y + 2/3, x + 1/3, -z + 1/3$	28: $-y + 2/3, -x + 1/3, z - 1/6$
29: $-x + y + 2/3, y + 1/3, z - 1/6$	30: $x + 2/3, x - y + 1/3, z - 1/6$
31: $-x + 1/3, -y + 2/3, -z + 2/3$	32: $y + 1/3, -x + y + 2/3, -z + 2/3$
33: $x - y + 1/3, x + 2/3, -z + 2/3$	34: $-y + 1/3, -x + 2/3, z + 1/6$
35: $-x + y + 1/3, y + 2/3, z + 1/6$	36: $x + 1/3, x - y + 2/3, z + 1/6$

Table 1c. Anisotropic displacement parameters ($\text{Å}^2 \times 10^3$) for

Atom	U11	U22	U33	U23	U13	U12
S	57(1)	45(1)	57(1)	2(1)	5(1)	28(1)
C	44(2)	43(2)	60(3)	-2(1)	-3(2)	22(1)
N	88(2)	59(2)	69(2)	-5(1)	1(1)	47(2)

The anisotropic displacement factor exponent takes the form $2\pi^2 [h^2 a^2 U(11) + \dots + 2hka^* b^* U(12)]$.

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