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The Experimental Charge Distribution in Sulfur-Containing Molecules. A Study of the Deformation Density in NH_4SCN at 81 K by Combined X-ray and Neutron Diffraction*

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The crystal structure of NH_4SCN has been redetermined at 81 K by an accurate X-ray and neutron diffraction study. The CS and CN bond lengths are 1·649 (1) and 1·176 (1) Å. The intermolecular bonding consists of two N—H···N and two N—H···S hydrogen bonds of intermediate strength. The deformation density in the SCN group is very similar to results obtained for NaSCN. Deformation density maps through the hydrogen bonds support the electrostatic nature of the hydrogen bond. The charge transfer from the NH_4 group towards the SCN group has been determined as 0·5–0·6 e.

Introduction

A determination of the electron density distribution in NaSCN by X-ray diffraction is described in the preceding paper (Bats, Coppens & Kvick, 1977). As possible differences between the electron density distribution in related structures are of interest, a similar study of NH_4SCN was undertaken.

Since the ammonium group contains four H atoms whose parameters cannot be obtained from the X-ray experiment, accurate neutron data are essential for the present study. A temperature of 81 K was selected for the combined X-ray and neutron experiment. This temperature can be reached in both experiments.

The room-temperature structure of NH_4SCN has previously been refined by Zavodnik, Zvonkova, Zhdanov & Mirevick (1972). No parameters for the H atoms were reported in that work, so that a further point of interest in the present study is the elucidation of the hydrogen-bond system in the NH_4SCN crystals.

Experimental

Neutron diffraction

Neutron diffraction data were collected at the high-flux beam reactor at Brookhaven National Laboratory. A parallelepiped-shaped crystal, 2·1 × 1·6 × 0·8 mm, was obtained from a methanol solution and mounted in a liquid-nitrogen-cooled cryostat. A thermocouple inside the cryostat recorded a temperature of 81·4 ± 0·1 K during the entire experiment. A calibration of the thermocouple using the ferromagnetic phase transition of an FeF_2 single crystal, mounted in the same way as the NH_4SCN specimen, indicated that the actual crystal temperature is within 0·8° of the recorded temperature.

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Crystallographic information for NH_4SCN at 81 K and data collection details are reported in Table 1. Data were collected on a four-circle diffractometer in two different quadrants of reciprocal space, up to a sin θ/λ value of 0·70 Å^{-1} with a wavelength of 1·0164 Å.

Two standard reflections recorded after every 50 reflections showed no significant fluctuations in intensity. The method of Blessing, Coppens & Becker (1974) was used to analyze the reflection profiles. Absorption corrections were calculated by a numerical integration with 216 Gaussian grid points and a linear absorption coefficient $\mu = 1{\cdot}47$ cm^{-1}. This includes an incoherent absorption cross-section for H of 35 b. Symmetry-related reflections were averaged to give 1042 independent reflections with net intensity larger than zero. The internal agreement between symmetry-equivalent reflections measured $\Sigma\,|F^2 - \langle F^2\rangle\,|\,/\Sigma\,F^2 = 1{\cdot}9\%$. A weighting scheme according to $w(F^2) = [\sigma(F^2)_{\rm counts} + 0{\cdot}02F^2]^{-2}$ described the variations between symmetry-related reflections adequately and was used in subsequent refinements.

X-ray diffraction

A carefully selected single crystal, 0·40 × 0·40 × 0·60 mm, grown from a solution in methanol was mounted in the cryostat described by Coppens, Ross, Blessing, Cooper, Larsen, Leipoldt, Rees & Leonard (1974) which is designed for the Picker diffractometer. Crystallographic information is reported in Table 1.

Data were collected at 81 K in three quadrants of reciprocal space up to respective sin θ/λ values of 0·75, 0·75 and 0·65 Å^{-1} with Nb-filtered Mo $K\alpha$ radiation. A step-scanning mode was used (Blessing, Coppens & Becker, 1974). An additional 390 reflections were measured in the range $0{\cdot}75 < \sin\theta/\lambda < 0{\cdot}95$ Å^{-1} at which point data collection had to be terminated because of equipment failures.

Lorentz, polarization and absorption factors were applied, and deviations from counter linearity corrected, as described by Chipman (1969). Averaging of symmetry-related reflections resulted in 1614 unique reflections with net intensity larger than zero ($\Sigma\,|\,F^2 - \langle F^2\rangle\,|\,/\Sigma\,F^2 = 0{\cdot}96\%$) which were assigned weights according to $w(F^2) = [\sigma^2(F^2)_{\rm counts} + (0{\cdot}015F^2)^2 + 10^2]^{-1}$.

Table 1. *General information*

	Neutron	X-ray
a	4·142 (1) Å	4·138 (2) Å
b	7·063 (3)	7·074 (3)
c	13·078 (3)	13·090 (5)
β	97·19 (1)°	97·19 (3)°
V	379·6 (2) Å^3	380·2 (3) Å^3
Space group	$P2_1/c$	$P2_1/c$
Wavelength	1·0164 Å	0·71069 Å
$(\sin\theta/\lambda)_{\rm max}$	0·70 Å^{-1}	0·75 Å^{-1}*
Total number of reflections	2716	3679
Number of unique reflections	1070	1644
Crystal volume	3·00 mm^3	0·11 mm^3
μ	1·47 cm^{-1}	5·95 cm^{-1}
Transmission range	0·789–0·891	0·747–0·796

* With some data at higher angles. See text.

Structure refinement

Neutron data

A Fourier synthesis based on the non-hydrogen atom parameters of Zavodnik, Zvonkova, Zhdanov & Mirevick (1972) showed the positions of all four H atoms. A refinement with scattering lengths $b_S = 0{\cdot}2847$, $b_C = 0{\cdot}6626$, $b_H = -0{\cdot}3723$ (Shull, 1971) and $b_N = 0{\cdot}920 \times 10^{-12}$ cm (Kvick, Koetzle, Thomas & Takusagawa, 1974) gave the results reported in Tables 2 and 3.* The isotropic extinction treatment (Becker & Coppens, 1974) indicated a mosaic spread of 11 seconds (Lorentzian distribution), and no significant anisotropy of the extinction. All refinements reported in this paper were based on F.

X-ray data

The refinement was started with the neutron parameters. Scattering factors were as listed in *International Tables for X-ray Crystallography* (1974), except those for H which were as given by Stewart, Davidson & Simpson (1965). For the S atom the anomalous dispersion factors of Cromer & Liberman (1970) were applied.

Separate least-squares refinements were performed on all X-ray data (conventional refinement) and on the

Table 2. *Review of least-squares refinements*

	Neutron	X-ray (conventional)	X-ray (high order)
$(\sin\theta/\lambda)_{\rm min}$	0·00 Å^{-1}	0·00 Å^{-1}	0·60 Å^{-1}
$(\sin\theta/\lambda)_{\rm max}$	0·70	0·75 †	0·75 †
Number of observations	1023	1614	932
Number of variables	74	54	36
Scale factor	114·7 (2)	13·29 (3)	fixed
Mosaic spread	11	26	fixed
$R(F)$	1·8%	2·0%	1·6%
$R_w(F)$	1·8	3·2	1·6
Goodness of fit*	0·98	3·17	1·40

* Defined as $[\,\Sigma\,w(|F_o| - |F_c|)^2/(\mathrm{NO} - \mathrm{NV})]^{1/2}$.
† With some data at higher angles. See text.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32244 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

X-ray data with $\sin \theta/\lambda > 0·60$ Å^{-1} (high-order refinement). In the latter refinement the scale factor and the H atom and extinction parameters had to be fixed at the full-data values because of the limited dependence of these parameters on the high-order data. Relevant information and results of both refinements are included in Tables 2 and 3.*

Table 3. *Positional and thermal* (×10^5) *parameters*

N: neutron refinement; *X*1: conventional X-ray refinement; *X*2: high-order X-ray refinement.

		x	*y*	*z*
S	*N*	−0·01141 (18)	0·98944 (10)	0·19465 (5)
	*X*1	−0·01133 (4)	0·98933 (2)	0·19474 (1)
	*X*2	−0·01125 (3)	0·98933 (1)	0·19474 (1)
C	*N*	0·07894 (8)	0·82576 (4)	0·11331 (2)
	*X*1	0·07912 (17)	0·82559 (9)	0·11323 (5)
	*X*2	0·07905 (13)	0·82585 (5)	0·11336 (3)
N(1)	*N*	0·14395 (6)	0·70831 (3)	0·05579 (2)
	*X*1	0·14412 (17)	0·70840 (9)	0·05585 (5)
	*X*2	0·14425 (14)	0·70817 (6)	0·05569 (3)
N(2)	*N*	0·45231 (6)	0·33889 (3)	0·11277 (2)
	*X*1	0·45224 (16)	0·33878 (9)	0·11289 (5)
	*X*2	0·45240 (12)	0·33875 (5)	0·11285 (3)
H(1)	*N*	0·58622 (20)	0·30058 (12)	0·05479 (6)
	*X*1	0·5649 (31)	0·3056 (19)	0·0611 (10)
H(2)	*N*	0·60886 (20)	0·38058 (13)	0·17620 (6)
	*X*1	0·5925 (29)	0·3759 (19)	0·1703 (10)
H(3)	*N*	0·31512 (21)	0·22656 (11)	0·13286 (7)
	*X*1	0·3325 (33)	0·2356 (20)	0·1287 (9)
H(4)	*N*	0·30343 (20)	0·45089 (11)	0·08876 (6)
	*X*1	0·3139 (30)	0·4349 (20)	0·0916 (10)

Discussion of the structure

Bond distances and angles are reported in Table 4, while a stereoscopic view of the structure is shown in Fig. 1.

The CN and CS bond lengths agree within the standard deviations with identical bonds observed in NaSCN (Bats, Coppens & Kvick, 1977); while the SCN angle in NH_4SCN is slightly larger than in NaSCN (179·60 ± 0·04° against 179·09 ± 0·04° in NaSCN).

Table 4. *Bond lengths* (Å) *and angles* (°)

The bond length values in square brackets are corrected for libration according to a rigid-body model for the SCN group.

	Neutron	X-ray (full data)	X-ray (high order)
S—C	1·6456 (9)	1·6493 (8)	1·6467 (6)
	[1·649]	[1·653]	[1·650]
C—N(1)	1·1736 (5)	1·1723 (10)	1·1776 (6)
	[1·176]	[1·175]	[1·180]
N(2)—H(1)	1·0301 (8)	0·90 (1)	–
N(2)—H(2)	1·0296 (8)	0·93 (1)	–
N(2)—H(3)	1·0291 (9)	0·92 (1)	–
N(2)—H(4)	1·0276 (8)	0·91 (1)	–
S—C—N(1)	179·60 (4)	179·54 (6)	179·57 (4)
H(1)—N(2)—H(2)	109·00 (8)	111 (1)	–
H(1)—N(2)—H(3)	110·53 (8)	108 (1)	–
H(1)—N(2)—H(4)	109·60 (7)	109 (1)	–
H(2)—N(2)—H(3)	109·10 (8)	110 (1)	–
H(2)—N(2)—H(4)	108·59 (8)	110 (1)	–
H(3)—N(2)—H(4)	109·98 (8)	109 (1)	–

Table 3 (*cont.*)

		U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S	*N*	1221 (31)	1020 (32)	1177 (31)	202 (24)	347 (24)	−21 (24)
	*X*1	1287 (15)	1017 (8)	1176 (9)	232 (5)	313 (8)	−34 (5)
	*X*2	1272 (6)	1026 (3)	1164 (3)	225 (3)	317 (4)	−24 (2)
C	*N*	1244 (14)	1030 (15)	981 (13)	159 (10)	235 (10)	−42 (10)
	*X*1	1081 (32)	1068 (22)	1076 (23)	54 (23)	105 (23)	138 (18)
	*X*2	1236 (19)	1030 (11)	1048 (11)	136 (13)	188 (13)	17 (9)
N(1)	*N*	1900 (12)	1358 (12)	1278 (11)	322 (9)	321 (8)	−290 (8)
	*X*1	1872 (34)	1423 (23)	1334 (23)	246 (24)	218 (23)	−157 (18)
	*X*2	1938 (21)	1400 (12)	1326 (12)	323 (14)	288 (14)	−255 (10)
N(2)	*N*	1208 (11)	1290 (13)	1136 (11)	163 (8)	202 (8)	38 (8)
	*X*1	1254 (31)	1270 (22)	1210 (23)	155 (22)	172 (22)	56 (17)
	*X*2	1236 (18)	1277 (11)	1199 (11)	149 (12)	173 (12)	46 (9)
H(1)	*N*	3025 (37)	3403 (39)	2546 (35)	494 (31)	1134 (29)	−326 (29)
	*X*1	2530 (300)					
H(2)	*N*	3041 (37)	3570 (39)	2284 (33)	−197 (32)	−443 (29)	−289 (30)
	*X*1	2730 (320)					
H(3)	*N*	3214 (39)	2688 (38)	3712 (41)	−723 (32)	823 (32)	435 (32)
	*X*1	3010 (340)					
H(4)	*N*	2916 (36)	2868 (38)	3261 (38)	1150 (31)	423 (30)	580 (30)
	*X*1	2450 (310)					

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* See deposition footnote on p. 1543.

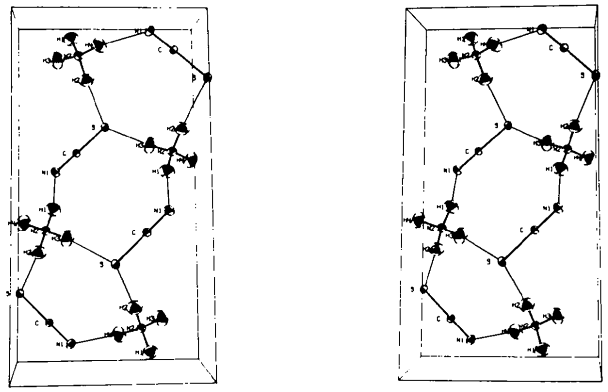


Fig. 1. Stereoscopic view of the structure. The thermal ellipsoids are 50% probability surfaces.

The main intermolecular bonding is formed by four hydrogen bonds, two of the type N—H···N and two of type N—H···S, listed in Table 5. Both H···N and H···S interactions are about 0·4 Å shorter than the sum of their van der Waals radii; all four hydrogen bonds can therefore be classified as being of intermediate strength.

The thermal parameters of the atoms of the SCN group are largest in the direction perpendicular to the molecular axis. This is especially true for N(1), which is furthest from the center of libration found to be in the CS bond at a distance of approximately 0·75 Å from the C atom. This compares well with the center of mass of a free SCN group (in the CS bond at 0·62 Å from the C atom). Very similar results have been obtained for the SCN group in NaSCN (Bats, Coppens & Kvick, 1977).

Analyses of chemical bonding

Effect on atomic parameters

In order to analyze the influence of the bonding electrons on the positional parameters obtained from the conventional and high-order X-ray refinements, asphericity shifts of the atomic positions with respect to the neutron parameters are reported in Table 6. They are very small (about 0·002 Å), but systematic: in the conventional X-ray refinement the C atom undergoes a small shift into the almost triple CN bond, while in the high-order X-ray refinement the N(1) atom shows a small shift into its lone-pair direction. Similar though larger shifts have been observed in tetracyanoethylene (Little, Pautler & Coppens, 1971; Becker, Coppens & Ross, 1973). The small lengthening of the CN bond has also been observed in the high-order X-ray refinement of NaSCN (Bats, Coppens & Kvick, 1977) and implies that the nitrogen lone-pair density scatters beyond the sin θ/λ limit of 0·60 Å^{-1}. Electron density studies based on X-ray data alone may therefore underestimate the deformation density in the cyano group.

As expected, high-order thermal parameters are in better agreement with the neutron results than the full-data thermal parameters.

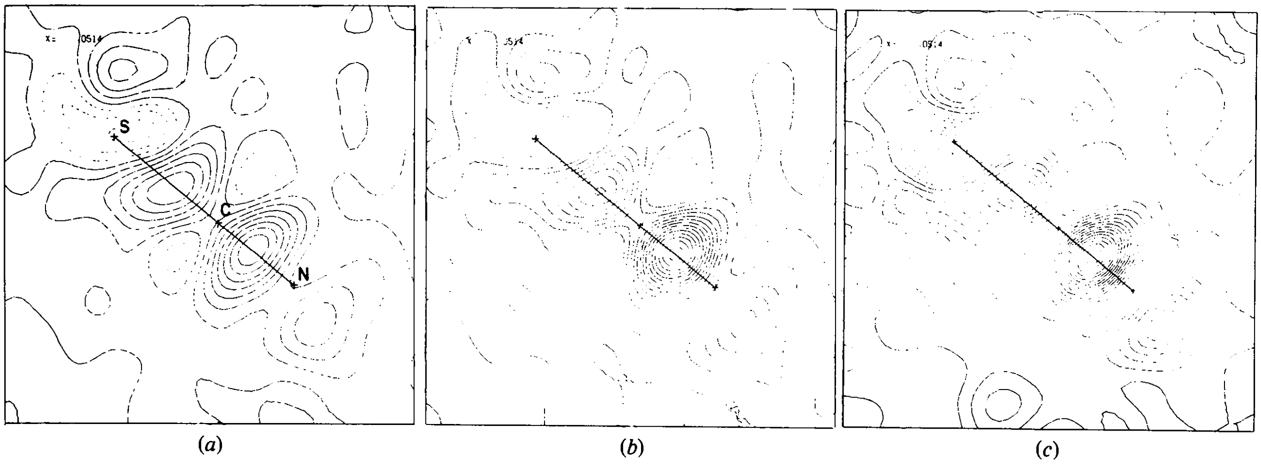
There are often systematic differences between X-ray and neutron thermal parameters, even when high-order X-ray refinements are available (Bats, Coppens & Koetzle, 1977), but in the present study the ratio's U_{ii} (neutron)/U_{ii} (high-order X-ray) averaged over all non-hydrogen atoms are 0·985 (10), 0·998 (10) and 0·958 (9) for U_{11}, U_{22} and U_{33} respectively. Though the U_{33} values are slightly smaller in the neutron study than in the high-order X-ray study, the agreement between the two sets is good.

Table 5. *Hydrogen bonds* (*neutron parameters*)

N–H···X	N···X(Å)	H···X(Å)	N–H–X(°)
N(2)–H(2)···S	3·373 (1)	2·350 (1)	172·47 (8)
N(2)–H(3)···S	3·383 (1)	2·358 (1)	173·67 (8)
N(2)–H(1)···N(1)	2·948 (1)	1·936 (1)	166·53 (7)
N(2)–H(4)···N(1)	2·959 (1)	1·964 (1)	162·19 (8)

Table 6. *Asphericity shifts of atomic positions* (Å)

	X (full data) – N	X (high order) – N	X (full data) – X (high order)
S	0·0019 (7)	0·0021 (7)	0·0004 (2)
C	0·0019 (5)	0·0011 (5)	0·0030 (7)
N(1)	0·0012 (5)	0·0021 (5)	0·0027 (7)
N(2)	0·0017 (5)	0·0014 (5)	0·0010 (7)
H(1)	0·13 (1)	–	–
H(2)	0·10 (1)	–	–
H(3)	0·11 (1)	–	–
H(4)	0·12 (1)	–	–



(a) (b) (c)

Fig. 2. Deformation density maps in a section through the thiocyanate group, based on atomic parameters from (a) full-data X-ray refinement, (b) high-order X-ray refinement, (c) neutron refinement. Data cut-off in Fourier synthesis: sin θ/λ = 0·65 Å^{-1}. Contour interval 0·05 e Å^{-3}; negative contours broken.

Table 7. *Observed peak heights in the* SCN *group* (e Å^{-3})

The values are based on a Fourier synthesis with data cut-off 0·75 Å^{-1}. Standard deviations are estimated at 0·04 e Å^{-3}.

	NH_4SCN 81 K			NaSCN 81 K
	$X-X$ (full data)	$X-X$ (high order)	$X-N$	$X-X$ (high order)
C–N bond	0·37	0·66	0·76	0·70
C–S bond	0·31	0·38	0·40	0·28
N lone pair	0·19	0·23	0·32	0·26
S lone pair	0·18	0·17	0·15	0·15

Electron density maps

Deformation electron densities in the SCN plane based on the atomic parameters from the full-data X-ray, high-order X-ray and neutron refinements are compared in Fig. 2. In this order the maps show the deformation density with increasing detail. Maximum peak heights increase accordingly in this direction (Table 7). The difference between the high-order X-ray and neutron parameter maps can be attributed to the X-ray asphericity shifts, especially in the vicinity of the atom N(1).

The SCN density in NH_4SCN is in good agreement with results on NaSCN at the same temperature (Bats, Coppens & Kvick, 1977). A comparison of peak heights in the two structures is included in Table 7. The agreement supports the validity of this type of electron density measurement and indicates that the effect of the crystal environment on the density of the SCN^- ion is limited.

The deformation density in cross-sections through the CN and CS bonds is shown in Fig. 3. The CN bond population shows the expected cylindrical symmetry,

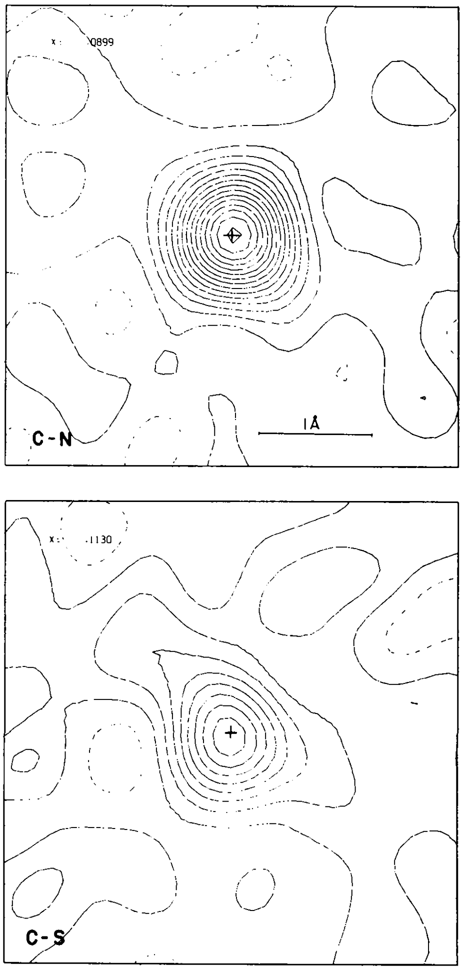


Fig. 3. X–N deformation density sections through the CN and CS bond peaks, normal to the bonds. Data cut-off and contours as in Fig. 2.

while deviations from the cylindrical appearance of the CS bond appear marginally significant.

The S lone-pair density appears in both SCN structures at the sides rather than at the back of the atom. Its cross-section perpendicular to the bond, shown in Fig. 4, is irregular. But the lone-pair is so low that any possible effects of the NH···S hydrogen bonding are masked by the limitations of the experimental accuracy.

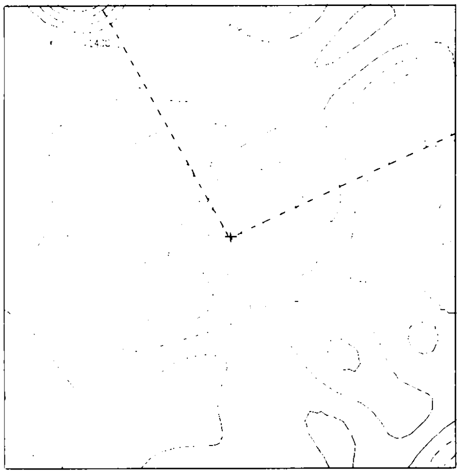


Fig. 4. Deformation density section normal to the CS bond, through the sulfur lone-pair density. Data cut-off and contours as in Fig. 2.

Sections through the four hydrogen bonds are shown in Fig. 5. As found in α-glycylglycine (Griffin & Coppens, 1975) and sulfamic acid (Bats, Coppens & Koetzle, 1977) there is no build-up of electron density in the intermolecular regions, in support of the electrostatic nature of the hydrogen bond. It should be noted that the NH group does not point directly towards the N or S lone-pair density in all four bonds. As pointed out by Almlöf, Kvick & Thomas (1973), this is not necessarily an indication of the weakness of the hydrogen bonds, as the electrostatic potential of the electronegative atoms has less directional character than the lone-pair electron density.

Charge transfer

In order to determine the charge transfer between the ammonium and thiocyanate group, the numerical integration method developed by Coppens (1975) has

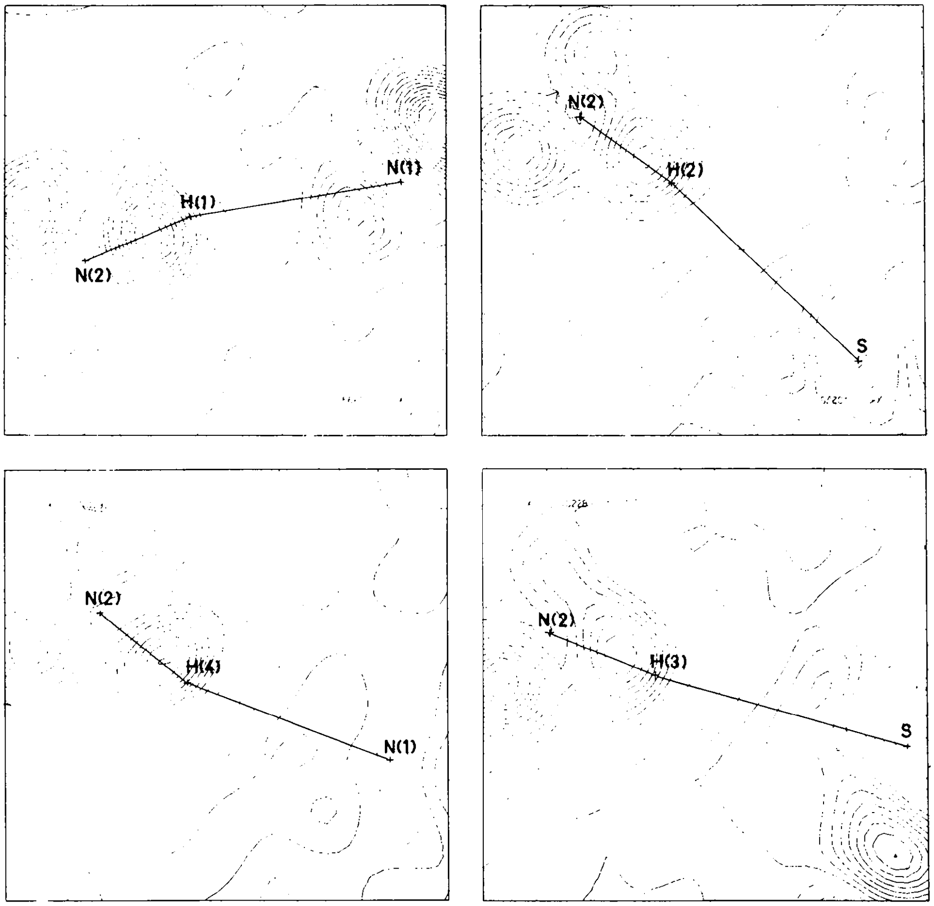


Fig. 5. $X-N$ deformation density sections through the hydrogen bonds. Data cut-off and contours as in Fig. 2.

been applied to the present data set. Application of this method to $NaCN.2H_2O$ (Bats, 1977) and NaSCN (Bats, Coppens & Kvick, 1977) showed the actual charge transfer between cation and anion to be considerably less than predicted by a pure ionic model.

The integration has been performed over the deformation density with the integration boundaries for the NH_4 and SCN group defined by the ratio of van der Waals radii as described by Coppens (1975). With van der Waals radii for S, C, N and H of 1·85, 1·50, 1·50 and 1·1 Å respectively, a charge transfer of 0·53 (4) e from the ammonium group towards the thiocyanate group was obtained. The standard deviation for this value is based on the experimental errors in the observed structure factors and on an error in the scale factor of 1%, which seems a reasonable value for the present study. An increase of the volume assigned to the NH_4 group leads to an increase in charge transfer up to 0·65 (8) e which may be considered an upper limit. Though this charge transfer (0·5–0·6 e) is considerably smaller than the value of 1·0 e for a pure ionic model, it is larger than the result of 0·27 (6) e obtained for NaSCN. The difference implies a larger contribution of electrostatic bonding between cation and anion in NH_4SCN.

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