Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.

Refinement of the crystal structure of 2,2′,2″-triaminotriethylaminenickel(II) dithiocyanate. By P.D.CRAD-WICK and D.HALL, *Department of Chemistry, University of Alberta, Edmonton, Alberta, Canada.*

The data sets from two previous structure determinations of 2,2′,2″-triaminotriethylaminenickel(II) dithio-cyanate have been merged, and the structure refined by least squares.

The crystal structure of 2,2′,2″-triaminotriethylamine-nickel(II) dithiocyanate has been determined independently by Rasmussen (1958, 1959) and by Hall & Woulfe (1958). The configuration about the metal is octahedral, with the ligand quadridentate through the four nitrogen atoms, and with the two anions also coordinated through the nitrogen atoms, as in Fig. 1.

The two analyses led to essentially the same structure but each was based on somewhat limited data – Rasmussen's data were three-dimensional but contained only 712 of mainly the more intense, low-angle reflexions, whereas Hall & Woulfe's data comprised only the three principal projections, although the 383 terms did include the majority of such data accessible with Cu $K\alpha$ radiation. The errors in both analyses were consequently high, and the reported bond lengths showed some large deviations from normal values.

It appeared that the two data sets were to some extent complementary, and that a combination of the two might be sufficient to yield a more satisfactory structure. Accordingly they were scaled together, using the least-squares procedure of Rae (1965), yielding a new set of 867 non-zero terms. The common reflexions showed a mean discrepancy in F of 15%. The parameters listed by Rasmussen were then refined by full-matrix least-squares, assuming isotropic temperature factors, to an R index of 0·127, and on the assumption of anisotropic parameters for the nickel and sulphur atoms, to 0·107. Scattering factors were as in *International Tables for X-ray Crystallography* (1962), with a real dispersion correction for nickel; the function minimized was $\sum w(F_o - F_c)^2$, where $w = a^2/\{a^2 + (F-b)^2\}$, a and b being chosen so that $\langle w|F_o - F_c|\rangle$ was invariant with $|F|$. A further attempt at refinement by allowing anisotropic parameters for the lighter atoms led to these assuming

Table 1. *Atomic parameters*

(*a*) Atom coordinates

	x/a	y/b	z/c	B
Ni	0·1220 (3)	0·3333 (3)	0·8347 (4)	1·38 Å²
S(1)	0·0082 (7)	0·0209 (4)	0·7600 (9)	3·23
S(2)	−0·3081 (5)	0·3868 (5)	0·9310 (8)	2·59
C(1)	−0·169 (2)	0·359 (2)	0·885 (2)	1·3 (4)
C(2)	0·077 (2)	0·119 (2)	0·785 (3)	2·0 (4)
C(3)	0·268 (2)	0·363 (2)	0·548 (3)	2·3 (4)
C(4)	0·353 (2)	0·315 (2)	0·672 (3)	1·9 (4)
C(5)	0·295 (2)	0·296 (2)	1·101 (3)	2·6 (5)
C(6)	0·361 (2)	0·276 (2)	0·950 (3)	2·1 (4)
C(7)	0·255 (2)	0·497 (2)	0·947 (3)	2·4 (4)
C(8)	0·351 (2)	0·444 (2)	0·864 (3)	1·9 (4)
N(1)	−0·065 (2)	0·348 (2)	0·858 (2)	2·3 (4)
N(2)	0·112 (2)	0·191 (2)	0·791 (3)	3·2 (4)
N(3)	0·140 (2)	0·384 (2)	0·593 (2)	2·3 (4)
N(4)	0·158 (2)	0·304 (2)	1·076 (2)	2·4 (4)
N(5)	0·130 (2)	0·477 (1)	0·885 (2)	2·5 (4)
N(6)	0·317 (2)	0·348 (2)	0·856 (2)	1·7 (3)

(*b*) Anisotropic thermal parameters

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0·012 (1)	0·021 (2)	0·020 (2)	0·002 (2)	−0·001 (2)	−0·005 (2)
S(1)	0·051 (4)	0·027 (4)	0·045 (4)	−0·009 (4)	−0·030 (4)	0·014 (4)
S(2)	0·019 (3)	0·035 (4)	0·045 (4)	0·014 (3)	0·009 (3)	0·016 (4)
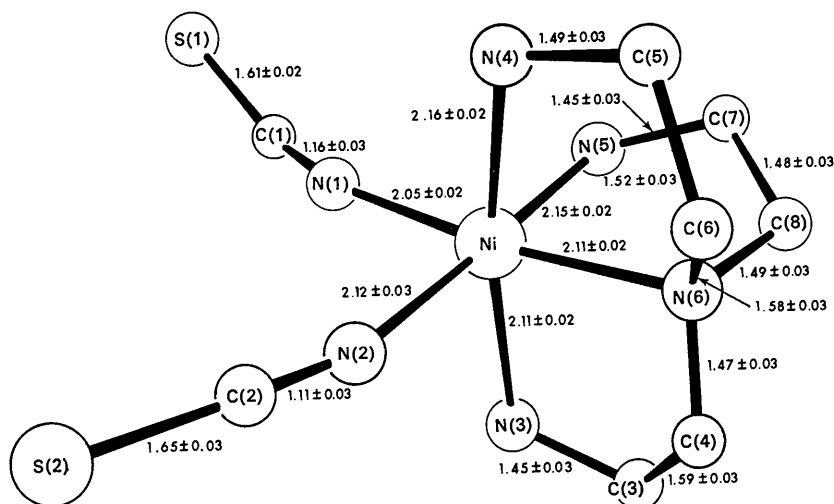
Fig. 1. Bond lengths.

Table 2. *Bond angles*

N(4)–Ni —N(5)	89·8 (8)°		Ni —N(6)–C(8)	110 (1)°
N(4)–Ni —N(2)	89·0 (8)		Ni —N(6)–C(4)	105 (1)
N(4)–Ni —N(1)	95·4 (8)		Ni —N(2)–C(2)	162 (2)
N(4)–Ni —N(6)	82·9 (7)		N(2)–C(2)–S(2)	171 (2)
N(5)–Ni —N(3)	93·3 (6)		Ni —N(1)–C(1)	169 (2)
N(5)–Ni —N(6)	82·2 (8)		N(1)–C(1)–S(1)	177 (2)
N(5)–Ni —N(1)	87·8 (9)		Ni —N(4)–C(5)	109 (2)
N(2)–Ni —N(3)	86·1 (8)		N(4)–C(5)–C(6)	111 (2)
N(2)–Ni —N(6)	98·6 (8)		C(5)–C(6)–N(6)	108 (2)
N(1)–Ni —N(3)	100·5 (8)		Ni —N(5)–C(7)	107 (2)
N(6)–Ni —N(3)	82·1 (7)		N(5)–C(7)–C(8)	112 (2)
C(6)–N(6)–C(8)	114 (2)		C(7)–C(8)–N(6)	115 (2)
C(6)–N(6)–C(4)	108 (2)		Ni —N(3)–C(3)	112 (1)
C(8)–N(6)–C(4)	117 (2)		N(3)–C(3)–C(4)	108 (2)
Ni —N(6)–C(6)	102 (1)		C(3)–C(4)–N(6)	108 (2)

physically unreal values, and was not continued. Atom coordinates corresponding to the above *R* index of 0·107 are listed in Table 1, bond lengths are shown in Fig. 1, and bond angles in Table 2.

The standard deviations are still high, but with the one exception of the length C(6)–N(6) all dimensions are now within error of expected values, and the combined data set has given a much more satisfactory picture of the molecule than did either separately.

References

HALL, D. & WOULFE, M. D. (1958). *Proc. Chem. Soc.* p. 346.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

RAE, A. D. (1965). *Acta Cryst.* **19**, 683.

RASMUSSEN, S. E. (1958). *J. Inorg. Nuclear Chem.* **8**, 441.

RASMUSSEN, S. E. (1959). *Acta Chem. Scand.* **13**, 2009.

An X-ray study of L-isoleucine.

By B. KHAWAS, *Division of Agricultural Physics, Indian Agricultural Research Institute, New Delhi 12, India*

L-Isoleucine crystallized from aqueous solution is orthorhombic, space group $P222_1$. The cell-edge dimensions are $a = 13·89_6$, $b = 20·17_3$, $c = 5·35_9$ Å and the unit-cell contains eight molecules. From a close resemblance of the cell dimensions and space group to those of D-leucine, it is inferred that the molecules of L-isoleucine are stacked in double-layer units parallel to the *bc* plane with their chain length along the *a* axis.

The crystal structures of D-leucine (Möller, 1949), DL-leucine (Möller, 1949; Dawson & Mathieson, 1951), and D-isoleucine hydrochloride and hydrobromide (Trommel & Bijvoet, 1954) have been reported. Dawson & Mathieson, (1951) reported that DL-isoleucine is triclinic. Against the background of the above extensive studies and sufficient