A involving Br(4) or Br(5) is the Br(4)---Br(5) distance of 3.52 A between ions related by the *a* glide plane. This contact and the other short intermolecular distance are dependent upon the inclination of the Re_3 triangle to the (001) plane, but are not necessarily related to the size of the Br(4)Re(2)Br(5) angle. When the short intramolecular contacts are considered it is clear that a marked reduction in repulsive forces has been achieved by the closing of this angle. First we note that the distances Br(4)---Br(6) and Br(5)---Br(6) are virtually independent of the angle under consideration. This leaves the contacts Br(1)---Br(4) and Br(2)---Br(5) as the important factors. These two independent distances average 3.74 A, which, when compared with the Br(1)---Br(1′) distance of 3.31 A and the corresponding 3.38 A in Re_3Br_9, indicates a considerable decrease in bromine–bromine repulsive forces across the face of the anion.

Accompanying the decrease in the Br(4)Re(2)Br(5) angle is a corresponding shortening of the two off-plane terminal bonds to a mean value of 2.38 A, compared with the mean value of 2.47 A for the bonds to Re(1). This difference of 0.09 A is about 7σ and therefore highly significant. It may be accounted for by postulating some π bonding between filled 3d orbitals of the terminal bromines and the rhenium 5p or 5s5p hybrid lying in the plane of the Re_3 triangle and unused in the absence of an in-plane terminal bromine. It is significant that shortening of the terminal off-plane bonds has not occurred in the isolated Re_3Br_9[10] species where *all* rhenium atoms are "deficient." Neither has the angle subtended by the pair of off-plane bromine atoms decreased. Additional π bonding cannot therefore be postulated in this case. It is hard to believe that intermolecular packing forces (which appear to be quite weak in the complex in which Re_3Br_9 occurs) could cause such wide variations as exist between the dimensions of $(Re_3Br_{11})^{2-}$ and neutral Re_3Br_9. For a full understanding of these variations we must await a quantitative bonding theory which considers the trinuclear cluster as a whole.

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CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY,
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The Crystal Structure of Chlorothiodiazyl Chloride, $S_3N_2Cl_2$[1]

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$S_3N_2Cl_2$ crystallizes in the monoclinic space group $P2_1$ with cell dimensions $a = 6.546$ A, $b = 8.600$ A, $c = 5.508$ A, and $\beta = 102.37°$; there are two formula units per unit cell. The material is a salt consisting of a chloride anion and a $\underline{\mathrm{S{-}N{-}S{-}N{-}S}}\mathrm{{-}Cl^+}$ cation. The sulfur and nitrogen atoms form a puckered five-membered ring.

Introduction

$S_3N_2Cl_2$ was first identified by Demarcay,[2] who prepared it by allowing SCl_2 or S_2Cl_2 to react with S_4N_4. Meuwsen[3] prepared $S_3N_2Cl_2$ by allowing $(NSCl)_3$ to react with S_2Cl_2. More recently Jolly, Maguire, and Rabinovich[4] produced the material by refluxing a suspension of ammonium chloride in S_2Cl_2. An X-ray crystal structure analysis on a crystal prepared by Jolly's method was undertaken to determine the molecular structure of this compound.

Experimental Section

$S_3N_2Cl_2$ is a yellow crystalline salt which decomposes in the air. The salt was removed from its container in a nitrogen-filled drybox, crushed, and sieved. Several crystal fragments of about 0.3 mm were introduced into tapered quartz capillaries of about the same size and sealed. With patient use of a microscope and a file, a single crystal fragment was isolated and wedged in the capillary; the capillary was fire sealed to a final length of about 1 cm. The crystal fragment was an irregular plate with dimensions approximately 0.3 × 0.4 × 0.2 mm. Some decomposition of the crystal surface did occur as the crystal lost its yellow color and took on a blackish tinge; however, the crystal diffracted X-rays well and no further decomposition was encountered. The crystal was oriented with the monoclinic *b* axis parallel to the length of the capillary.

A General Electric XRD-5 apparatus equipped with a quarter-circle Eulerian cradle type goniostat, a scintillation counter, a pulse height discriminator, and a molybdenum X-ray tube was used to measure the cell dimensions and the intensities. The X-ray tube was operated at 20 ma and 40 kv. The primary X-ray beam was filtered with a Zr filter.

The measured monoclinic cell dimensions based on $\lambda_{K\alpha_1} = 0.70926$ A are: $a = 6.546 \pm 0.007$ A, $b = 8.600 \pm 0.004$ A, $c = 5.508 \pm 0.005$ A, and $\beta = 102.37 \pm 0.03°$. The observed density was 2.0 g/ml, suggesting two formula units per unit cell;

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(1) Work done under the auspices of the U. S. Atomic Energy Commission.

(2) E. Demarcay, *Compt. Rend.*, **92**, 726 (1881).

(3) A. Meuwsen, *Ber.*, **65**, 1731 (1932).

(4) W. L. Jolly, K. D. Maguire, and D. Rabinovich, *Inorg. Chem.*, **2**, 1304 (1963).

the calculated X-ray density is 2.14 ± 0.01 g/ml. The observed extinctions are $0k0$, $k = 2n + 1$, and the space group is $P2_1$.

A total of 1022 independent reflections were each counted for 10 sec using a stationary crystal–stationary counter technique. These are all of the data in a quarter of the sphere of reflection where $2\theta < 62°$, or $\sin \theta/\lambda < 0.726$. Background was plotted as a function of 2θ and applied routinely to the reflections with the exception of a minority of intensities where very intense reflections would spill over on weaker ones in the same row; these backgrounds were then measured individually. Typical background counts were 20, 10, and 6 counts/sec at 20, 40, and 60° of 2θ, respectively. The most intense reflection ($10\bar{2}$) was about 56,000 counts/sec.

All of the computations were performed with an IBM 7044 computer with a 32K memory. The least-squares program used minimizes the function $\Sigma w(|F_o| - |F_c|)^2/\Sigma w F_o^2$, where w is the weighting factor and F_o and F_c are the observed and calculated structure factors. All of the reflections were given unit weights with the exception of the six most intense reflections which toward the end of the computations were given zero weight. Anisotropic temperature factors have the form $\exp -(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})$; however, for our convenience the program converts these thermal parameters to B_{ij} in A^2 where $B_{ij} = 4\beta_{ij}/a_i{}^*a_j{}^*$ and $a_i{}^*$ is the ith reciprocal axis length.

Atomic scattering factors[5] for neutral S, Cl, and N were used. S and Cl were both corrected for the anomalous dispersion effect, with $\Delta f' = 0.1$ and $\Delta f'' = 0.2$ electron.[6] The real component $\Delta f'$ was added to the scattering factors; the imaginary component $\Delta f''$ was incorporated into the actual least-squares calculation.

The "unreliability index" reported throughout this paper is $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$.

Determination of Structure

The structure was deduced from the three-dimensional Patterson function. A careful investigation of the function gave us trial positions for the three sulfur, two chlorine, and one nitrogen atoms; a Fourier based on the phases of the six-atom structure then gave the last nitrogen atom. As the atomic positions were deduced their parameters were introduced into a least-squares refinement using isotropic temperature factors. Starting with three atoms (Cl, S, N) the structure refined to $R = 0.47$, with four atoms (2 Cl, S, N) $R = 0.37$, with five atoms (2 Cl, 2 S, N) $R = 0.29$, with six atoms (2 Cl, 3 S, N) $R = 0.13$, and finally with all seven atoms $R = 0.09$. At the five-atom stage a nonexistent atom was introduced, but was rejected by the least squares *via* a large temperature parameter. The final identification of the sulfur and chlorine atoms was determined by studying the structure geometry and the interatomic distances. Anisotropic temperature factors were applied to all of the atoms and R reduced to 0.048. A few blunders in the data were detected, and these reflections were remeasured and corrected. Large discrepancies were present in the six most intense reflections which could be attributed to extinction effects and to the nonlinearity of the scintillation counter at high counting rates. These six reflections, all those with counting rates greater than 20,000 counts/sec, were assigned zero weight. The R decreased to 0.0292. The introduction of the imaginary dispersion correction increased R to 0.0293; the y parameters were all reversed and R became 0.0291. This difference is so small that there may be some doubt concerning which structure is correct, but we report the one which gave the best agreement. The other structure is almost identical except that the two nitrogen atoms are shifted 0.014 A toward zero along the y axis. The only bond lengths which are significantly different are S(1)–N(1) and S(1)–N(2), which are respectively longer and shorter by about 0.010 A in the other structure. This relative shift of light and heavy atoms along a polar direction when refined in this manner, or "polar dispersion shift," is an effect which has also been observed in thorium nitrate pentahydrate[7] and in a diiodocyclobutene[8] and which is explained in more detail elsewhere.[7] In the present case the ambiguity is of trivial importance to the chemical interpretation.

TABLE I

POSITIONAL[a] AND THERMAL[b] PARAMETERS IN $S_3N_2Cl_2$

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cl(1)	0.1017	0.0[c]	0.4844	3.32	5.33	3.63	1.18	1.51	1.24
Cl(2)	0.1975	−0.3541	0.1028	2.86	2.83	3.08	0.11	0.50	−0.09
S(1)	0.6099	0.1671	0.3235	2.37	3.48	2.81	0.37	0.37	−0.11
S(2)	0.2303	0.0264	0.1552	2.69	2.14	2.82	0.13	0.88	0.11
S(3)	0.2161	0.2734	0.1148	2.56	2.10	3.61	0.11	0.06	0.13
N(1)	0.4756	0.0105	0.2436	2.54	2.88	3.60	0.85	1.02	0.46
N(2)	0.4558	0.3039	0.2549	2.98	2.68	3.55	−0.10	0.30	−0.89

[a] Estimated standard deviations of the positional parameters for S and Cl are less than 0.0003, and for N they are less than 0.0008. [b] Estimated standard deviations of the thermal parameters for S and Cl are about 0.05, and for N they are about 0.2. Units of B_{ij} are A^2. [c] Polar space group $P2_1$ requires one y parameter to be fixed.

Results

A list of the atomic and thermal parameters of $S_3N_2Cl_2$ is shown in the Table I.

Table II presents a list of the observed and calculated structure factors.

The most interesting result of this work is the apparent ionic nature of the material. The material consists of a $S_3N_2Cl^+$ ion and a Cl^- ion. The chloride ion is Cl(2) and its closest approaches are to S(1) at 2.90 A, S(3) at 2.93 A, and S(2) at 3.04 A. Figure 1 shows a sketch of the $S_3N_2Cl^+$ ion labeled with distances and angles.

The three sulfurs and two nitrogens form a slightly

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(5) J. A. Ibers, "International Tables for X-ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962, p 202.

(6) D. H. Templeton, ref 5, p 215.

(7) T. Ueki, A. Zalkin, and D. H. Templeton, *Acta Cryst.*, **20**, 836 (1966).

(8) G. L. Hardgrove, L. K. Templeton, and D. H. Templeton, unpublished work.

TABLE II

OBSERVED AND CALCULATED STRUCTURE FACTORS (×10) OF $S_3N_2Cl_2$[a]

```
H,K=  0,  0
L FOBS FCAL
1  191  188
2   75   71
3  290  281
4  145  144
5  247  247
6   65   61
7   12    8

H,K=  0,  1
L FOBS FCAL
1  476  510
2  118  117
3  108  108
4  122  123
5   97   86
6   43   42
7   80   78

H,K=  0,  2
L FOBS FCAL
-0  434  466
1  293  302
2  129  129
3  253  258
4  187  186
5  125  122
6   87   87
7   12    5

H,K=  0,  3
L FOBS FCAL
1  331  349
2  454  480
3  203  205
4  146  147
5   35   33
6   87   82
7   64   64

H,K=  0,  4
L FOBS FCAL
-0  307  308
1  128  132
2   55   55
3  309  313
4   57   53
5   46   45
6   46   47
7   27   29

H,K=  0,  5
L FOBS FCAL
1  174  178
2  373  389
3  162  165
4   54   47
5   52   50
6   46   47
7   75   71

H,K=  0,  6
L FOBS FCAL
-0  345  346
1  115  116
2   58   58
3   37   36
4   50   50
5   97   96
6   80   81

H,K=  0,  7
L FOBS FCAL
1  179  182
2   20   22
3   78   79
4   25   25
5   60   60
6   29   24

H,K=  0,  8
L FOBS FCAL
-0  340  348
1  143  144
2   84   87
3  123  126
4  112  115
5   85   87

H,K=  0,  9
L FOBS FCAL
1   63   65
2   34   35
3   26   25
4   61   60
5   57   59

H,K=  0, 10
L FOBS FCAL
-0   35   37
1   60   62
2   15   15
3   50   57
4   60   61

H,K=  0, 11
L FOBS FCAL
1   73   75
2   90   95
3   47   53

H,K=  0, 12
L FOBS FCAL
-0   43   46
1   38   39
2   15   14

H,K=  1,  0
L FOBS FCAL
-7   65   66
-6   99   97
-5  104  101
-4  114  112
-3   22   21
-2  977 1140*
-1  468  473*
-0   28   24
1  428  440
2  212  217
3  513  538
4   63   56
5   12   15
6   93   89
7   49   44

H,K=  1,  1
L FOBS FCAL
-7   33   36
-6  104  101
-5   33   33
-4  169  168
-3  348  352
-2  106  107
-1  364  375
-0  175  162
1  319  312
2  123  124
3  182  183
4   89   92
5   80   91
6   61   63
7   24   30

H,K=  1,  2
L FOBS FCAL
-7   72   72
-6   92   91
-5  106  107
-4   33   33
-3  132  131
-2  321  320
-1  263  250
-0  243  238
1  328  342
2  281  278
3  218  217
4  156  154
5   18   17
6   59   56
7   80   78

H,K=  1,  3
L FOBS FCAL
-7   27   31
-6  111  112
-5  116  116
-4  206  204
-3  317  320
-2   55   54
-1  153  148
-0  691  776*
1  252  258
2   94   95
3   55   56
4  101  100
5  195  194
6   60   62
7   47   45

H,K=  1,  4
L FOBS FCAL
-7   33   34
-6   41   41
-5  160  159
-4   58   60
-3   53   53
-2  183  177
-1  158  151
-0  119  116
1  389  392
2   65   65
3   88   87
4   80   80
5   53   51
6  111  109
7   44   43

H,K=  1,  5
L FOBS FCAL
-7   15   11
-6   60   57
-5   59   59
-4  250  249
-3  192  194
-2   56   59
-1   99   95
-0  431  433
1  312  322
2   54   58
3  112  113
4   91   92
5  155  154
6   20   22

H,K=  1,  6
L FOBS FCAL
-6   47   45
-5   21   23
-4   66   65
-3  111  114
-2  262  259
-1   76   69
-0   48   47
1   43   29
2   54   54
3  168  166
4  120  123
5   45   46
6   25   21

H,K=  1,  7
L FOBS FCAL
-6   33   33
-5   51   54
-4   62   64
-3  162  163
-2   66   65
-1  127  122
-0   69   66
1   96  100
2   29   34
3   91   91
4   41   39
5   34   36
6   39   41

H,K=  1,  8
L FOBS FCAL
-6   54   49
-5   73   74
-4   53   51
-3   61   61
-2  284  287
-1  205  199
-0   94   92
1  134  131
2  183  183
3  170  173
4   15   13
5   46   47

H,K=  1,  9
L FOBS FCAL
-5   31   27
-4   16   13
-3   33   34
-2   49   50
-1   84   85
-0   43   39
1   45   42
2   56   54
3   75   74
4   50   52
5   20   18

H,K=  1, 10
L FOBS FCAL
-4   24   25
-3   34   37
-2   36   38
-1   67   63
-0   18   16
1   49   53
2   79   81
3   59   60
4   12   14

H,K=  1, 11
L FOBS FCAL
-3   67   69
-2   22   23
-1   54   54
-0  113  113
1   65   67
2   52   55
3   29   24

H,K=  1, 12
L FOBS FCAL
-2   34   37
-1   45   49
-0   17   13
1   40   40

H,K=  2,  0
L FOBS FCAL
-7    8    9
-6  103  101
-5    0    1
-4  403  407
-3  427  432
-2   53   43
-1  194  178
-0  349  349
1  726  776*
2  131  122
3   44   44
4  172  171
5  134  133
6   71   70
7   53   53

H,K=  2,  1
L FOBS FCAL
-7   23   21
-6  121  120
-5  174  172
-4   84   83
-3  197  196
-2  192  180
-1  368  355
-0  312  309
1  285  287
2  157  155
3   34   35
4  147  147
5   23   26
6   92   90
7   33   33

H,K=  2,  2
L FOBS FCAL
-7   14   17
-6   74   72
-5   41   39
-4  199  194
-3  120  112
-2  107  107
-1  141  138
-0  231  213
1  360  359
2  133  129
3   54   53
4  110  108
5  127  127
6   71   70
7   35   33

H,K=  2,  3
L FOBS FCAL
-7   39   39
-6   86   84
-5  186  183
-4   30   31
-3  130  129
-2  561  581
-1  242  236
-0  146  140
1  117  119
2   72   71
3  415  419
4  109  108
5   70   70
6   35   26
7   30   32

H,K=  2,  4
L FOBS FCAL
-7   50   51
-6   74   75
-5    7    3
-4   23   13
-3  134  130
-2  130  127
-1  251  238
-0   99   96
1  102   93
2  101  111
3   62   62
4  213  212
5   33   28
6   68   64

H,K=  2,  5
L FOBS FCAL
-7   12   13
-6  115  113
-5  127  127
-4   44   45
-3   62   60
-2  314  310
-1  384  392
-0  185  179
1  148  147
2  141  141
3  257  257
4   88   97
5   41   43
6   31   32

H,K=  2,  6
L FOBS FCAL
-7   18   19
-6   53   52
-5   79   77
-4  121  117
-3   86   83
-2   73   72
-1   59   57
-0   41   35
1  187  175
2  109  109
3   83   82
4   30   29
5   49   49
6   59   58

H,K=  2,  7
L FOBS FCAL
-6   58   59
-5   94   91
-4   74   74
-3   46   45
-2   87   86
-1  102  113
-0   91   88
1   88   86
2   63   63
3   57   58
4   62   64
5   22   25

H,K=  2,  8
L FOBS FCAL
-6   28   26
-5   36   38
-4  162  151
-3  173  175
-2   68   68
-1   76   72
-0  200  195
1  241  239
2   79   80
3   77   75
4   79   80
5   69   70

H,K=  2,  9
L FOBS FCAL
-5   15   16
-4   51   53
-3   70   71
-2   24   23
-1   59   55
-0   39   36
1   54   55
2  101  104
3   26   28
4   54   53

H,K=  2, 10
L FOBS FCAL
-4   32   34
-3   36   38
-2   46   44
-1   27   26
-0   71   69
1   84   82
2   21   19
3   25   25
4   23   28

H,K=  2, 11
L FOBS FCAL
-3   31   31
-2   90   92
-1   76   77
-0   62   64
1   18   23
2   49   48

H,K=  2, 12
L FOBS FCAL
-1   28   26
-0   55   55

H,K=  3,  0
L FOBS FCAL
-7   11    5
-6  108  109
-5  285  287
-4   43   44
-3   76   74
-2  311  307
-1  527  557
-0  429  416
1  110  107
2  193  178
3  318  313
4  141  140
5   76   75
6   12   10

H,K=  3,  1
L FOBS FCAL
-7   42   44
-6   62   62
-5   93   91
-4   58   53
-3  183  175
-2  263  253
-1  164  161
-0  184  178
1  133  128
2  204  207
3    6    9
4  193  189
5   59   60
6   65   64

H,K=  3,  2
L FOBS FCAL
-7   20   18
-6  122  121
-5   88   84
-4   17   17
-3  129  128
-2   90   83
-1  384  381
-0   81   71
1  117  112
2  111  109
3  123  118
4  156  153
5   49   46
6    8    9

H,K=  3,  3
L FOBS FCAL
-7   63   63
-6   13   11
-5   98   98
-4  264  260
-3  211  205
-2  149  144
-1   81   81
-0   83   78
1  562  580
2  166  162
3   86   86
4   99   97
5   49   49
6   83   90

H,K=  3,  4
L FOBS FCAL
-7   15   10
-6   50   49
-5   85   85
-4  107  107
-3   79   77
-2  146  144
-1   86   77
-0  185  173
1   61   61
2  269  264
3   71   75
4  111  111
5   44   46
6   39   39

H,K=  3,  5
L FOBS FCAL
-7   47   50
-6   21   22
-5   47   45
-4  154  152
-3  285  281
-2  187  184
-1   84   82
-0  146  139
1  329  322
2  184  181
3   94   94
4   70   73
5   59   54
6   65   60

H,K=  3,  6
L FOBS FCAL
-6   45   41
-5   85   85
-4   76   75
-3   98   98
-2   41   39
-1  119  109
-0   96   89
1  101  101
2   44   44
3   73   72
4   95   96
5   62   62

H,K=  3,  7
L FOBS FCAL
-6   47   48
-5    8    9
-4   42   44
-3   67   69
-2   94   94
-1   50   47
-0   83   79
1   85   35
2   56   59
3   27   26
4  106  104
5    9   16

H,K=  3,  8
L FOBS FCAL
-5  102  106
-4   60   50
-3    7    5
-2  144  142
-1  245  237
-0  152  149
1   61   63
2   99   97
3  138  136
4   47   47

H,K=  3,  9
L FOBS FCAL
-5   46   43
-4    0    4
-3   33   33
-2   37   37
-1   15   15
-0  121  119
1   35   36
2   45   46
3   50   50
4   26   29

H,K=  3, 10
L FOBS FCAL
-4   46   47
-3   12    9
-2   32   33
-1   91   91
-0   33   30
1   11   11
2   35   35
3   19   19

H,K=  3, 11
L FOBS FCAL
-3   62   63
-2   52   52
-1   15   15
-0   47   45
1   92   96
2   45   45

H,K=  4,  0
L FOBS FCAL
-7  125  129
-6   22   24
-5  107  107
-4  169  165
-3  273  268
-2  450  444
-1  125  123
-0   74   64
1  488  490
2  208  201
3   35   37
4   90   79
5   11    5
6   71   74

H,K=  4,  1
L FOBS FCAL
-7   25   24
-6   32   31
-5   68   65
-4  111  108
-3   34   34
-2  132  128
-1   99   96
-0  122  118
1   29   30
2  242  238
3   85   89
4   89   90
5   76   77
6    0    2

H,K=  4,  2
L FOBS FCAL
-7   41   42
-6    8    6
-5  123  122
-4   55   50
-3  275  267
-2  181  173
-1  125  118
-0   77   73
1   97   86
2  256  254
3   16   16
4   50   49
5   37   37
6   61   60

H,K=  4,  3
L FOBS FCAL
-7   37   41
-6   68   66
-5  161  157
-4   70   69
-3   31   31
-2   53   51
-1  451  453
-0  268  257
1   99  100
2  146  143
3   51   52
4  154  152
5   47   49
6    9   13

H,K=  4,  4
L FOBS FCAL
-7   27   29
-6   59   59
-5   67   66
-4  104  103
-3   87   83
-2  196  189
-1   87   89
-0  226  219
1  203  200
2  136  138
3   23   26
4   89   89
5   46   45

H,K=  4,  5
L FOBS FCAL
-7   28   28
-6   55   55
-5  143  143
-4  112  113
-3   21   23
-2   80   77
-1  290  283
-0  240  234
1   85   83
2   84   83
3   91   89
4  104  105
5   38   38

H,K=  4,  6
L FOBS FCAL
-6   40   43
-5   75   79
-4   45   42
-3   52   47
-2  117  115
-1   90   90
-0   78   80
1  113  108
2  103  101
3   67   69
4   39   37
5   50   54

H,K=  4,  7
L FOBS FCAL
-6   12    8
-5   30   32
-4   66   64
-3   42   42
-2   57   58
-1   58   59
-0   23   19
1   16   16
2  127  125
3   17   21
4   76   76

H,K=  4,  8
L FOBS FCAL
-5   22   22
-4   68   72
-3  173  167
-2  161  150
-1   21   19
-0   89   84
1  176  175
2  101   98
3   47   49
4   42   42

H,K=  4,  9
L FOBS FCAL
-5    9    3
-4   40   38
-3   18   14
-2   80   80
-1   36   33
-0   21   16
1   59   59
2   35   34
3   55   56

H,K=  4, 10
L FOBS FCAL
-4    9    9
-3   73   73
-2   32   35
-1   22   20
-0   44   38
1    8    6
2   74   76

H,K=  4, 11
L FOBS FCAL
-2   32   34
-1   81   83
-0   64   66

H,K=  5,  0
L FOBS FCAL
-7   51   50
-6   36   38
-5  126  122
-4  287  287
-3   51   51
-2   28   27
-1  441  443
-0  250  240
1   54   48
2  152  154
3   18   19
4  153  154
5   42   42

H,K=  5,  1
L FOBS FCAL
-7   28   28
-6   21   21
-5  102  103
-4   87   87
-3    9    9
-2   26   27
-1   58   59
-0  165  163
1  141  139
2  110  110
3   82   81
4   20   21
5   73   71

H,K=  5,  2
L FOBS FCAL
-7   56   57
-6   11   12
-5  135  137
-4  136  133
-3   48   44
-2   63   69
-1  104   98
-0  282  278
1   37   36
2   90   86
3   49   48
4   66   66
5   81   83

H,K=  5,  3
L FOBS FCAL
-7   81   83
-6   11   12
-5   69   69
-4   28   31
-3  211  207
-2  208  205
-1   89   90
-0   99   96
1   76   77
2  186  184
3   66   64
4   46   45
5   24   27

H,K=  5,  4
L FOBS FCAL
-7   36   37
-6   29   31
-5   53   55
-4  121  114
-3   87   84
-2  178  125
-1  227  275
-0  114  110
1   13   14
2  122  121
3   89   87
4   24   27
5   50   54

H,K=  5,  5
L FOBS FCAL
-6   47   51
-5   66   66
-4   40   41
-3  183  178
-2  215  213
-1   26   26
-0   53   45
1  105  105
2  153  151
3   63   63
4   50   49

H,K=  5,  6
L FOBS FCAL
-6   28   29
-5   23   24
-4   88   88
-3   49   49
-2   77   76
-1  107  100
-0   77   73
1   64   65
2   60   59
3   78   80
4   55   54

H,K=  5,  7
L FOBS FCAL
-5   48   52
-4   30   29
-3   11   10
-2   27   23
-1   25   23
-0   92   86
1   69   71
2   85   85
3   30   31
4   12   12

H,K=  5,  8
L FOBS FCAL
-5   77   79
-4  112  112
-3   14   16
-2   53   49
-1  149  151
-0  144  141
1   70   69
2   51   52
3   33   37

H,K=  5,  9
L FOBS FCAL
-4   31   31
-3   25   27
-2   12   16
-1   49   52
-0   32   32
1   66   65
2   12   10

H,K=  5, 10
L FOBS FCAL
-3   36   34
-2   28   28
-1    9   11
-0   70   71
1   20   18

H,K=  6,  0
L FOBS FCAL
-7   50   51
-6  114  117
-5   26   22
-4   26   25
-3  227  229
-2  243  243
-1   94   90
-0  145  144
1    7   12
2  210  209
3   86   88
4   26   28

H,K=  6,  1
L FOBS FCAL
-7   68   71
-6   41   39
-5   41   43
-4   76   76
-3   63   63
-2   71   69
-1  178  177
-0  111  112
1   30   33
2   59   59
3   93   93
4   44   44

H,K=  6,  2
L FOBS FCAL
-6   57   57
-5   33   37
-4   33   31
-3   52   51
-2  193  189
-1   50   49
-0   62   58
1   35   34
2   57   54
3  125  126
4   12   17

H,K=  6,  3
L FOBS FCAL
-6   14   21
-5   57   54
-4  235  237
-3   57   55
-2   20   21
-1  107  103
-0  151  145
1  120  121
2   81   83
3   27   27
4   51   56

H,K=  6,  4
L FOBS FCAL
-6   35   32
-5   65   66
-4   49   46
-3  119  135
-2   42   40
-1   22   18
-0   90   89
1  121  120
2   63   61
3   63   64
4   17   18

H,K=  6,  5
L FOBS FCAL
-6   24   26
-5   83   82
-4  145  144
-3   21   23
-2   42   42
-1   95   94
-0  175  172
1   90   93
2   60   59
3   12   16
4   37   39

H,K=  6,  6
L FOBS FCAL
-5    8   10
-4   38   37
-3   47   47
-2   61   58
-1   47   50
-0   53   51
1   82   82
2   60   60
3   62   62

H,K=  6,  7
L FOBS FCAL
-5    9   12
-4   37   29
-3   41   43
-2   35   28
-1   89   91
-0   64   64
1   21   18
2   19   22
3   42   44

H,K=  6,  8
L FOBS FCAL
-4   15   17
-3   86   85
-2  129  131
-1   62   63
-0   36   37
1   54   54
2   72   78

H,K=  6,  9
L FOBS FCAL
-3   43   40
-2   19   23
-1   47   49
-0   19   16
1   20   19

H,K=  7,  0
L FOBS FCAL
-6    0    3
-5   54   54
-4  186  190
-3   65   64
-2   49   52
-1   20   22
-0  175  177
1  150  152
2   29   29
3   68   71
4    0    2

H,K=  7,  1
L FOBS FCAL
-6   37   43
-5   33   32
-4   54   54
-3  130  134
-2   75   80
-1   45   45
-0   75   73
1   86   85
2   84   82
3   84   89

H,K=  7,  2
L FOBS FCAL
-6    9   14
-5    8    5
-4   87   90
-3   43   39
-2   23   20
-1   38   34
-0   43   42
1  116  119
2   25   28
3   28   24

H,K=  7,  3
L FOBS FCAL
-6  112  115
-5   14   15
-4   22   29
-3   77   78
-2   88   95
-1  154  158
-0   89   87
1   13   13
2   85   86
3   65   69

H,K=  7,  4
L FOBS FCAL
-5   44   43
-4   14   13
-3   11   10
-2   20   19
-1  113  118
-0   66   62
1   35   37
2   31   30
3   71   69

H,K=  7,  5
L FOBS FCAL
-5   26   28
-4   41   42
-3   55   59
-2  141  141
-1  104  117
-0   43   42
1   29   33
2   44   47

H,K=  7,  6
L FOBS FCAL
-4   55   56
-3   16   21
-2   20   17
-1   54   51
-0   32   35
1   63   61
2   55   59

H,K=  7,  7
L FOBS FCAL
-4    9    7
-3   64   64
-2   31   34
-1   12   12
-0   22   26
1   42   40

H,K=  7,  8
L FOBS FCAL
-2   21   23
-1   48   52
-0   71   73

H,K=  8,  0
L FOBS FCAL
-5   28   27
-4   20   19
-3   34   39
-2   89   89
-1  179  185
-0   33   29
1   68   67
2   24   29

H,K=  8,  1
L FOBS FCAL
-5   52   53
-4   31   29
-3   41   45
-2   37   40
-1   58   57
-0  104  107
1   76   81
2   32   34

H,K=  8,  2
L FOBS FCAL
-5   27   34
-4   56   59
-3   42   46
-2   33   31
-1   79   80
-0   38   41
1   14   10
2   31   29

H,K=  8,  3
L FOBS FCAL
-5   23   24
-4   47   48
-3  114  121
-2   42   43
-1   14    7
-0   86   86
1   71   73
2   29   32

H,K=  8,  4
L FOBS FCAL
-4   33   35
-3   75   80
-2   38   39
-1   16   17
-0   33   39
1   55   56

H,K=  8,  5
L FOBS FCAL
-4   75   77
-3   79   84
-2   12    7
-1   37   38
-0   45   48
1   83   87

H,K=  8,  6
L FOBS FCAL
-3   12   16
-2    9    1
-1   64   67
-0   53   59

H,K=  9,  0
L FOBS FCAL
-4   25   24
-3  142  150
-2   10   15
-1   35   37
-0   52   51

H,K=  9,  1
L FOBS FCAL
-4   20   21
-3   27   31
-2   71   74
-1   34   37
-0   31   37

H,K=  9,  2
L FOBS FCAL
-3   53   57
-2   33   38
-1   12   11
-0   36   36

H,K=  9,  3
L FOBS FCAL
-3   12   10
-2   46   49
-1   68   71
-0   34   37
```

[a] Asterisk indicates zero weight.

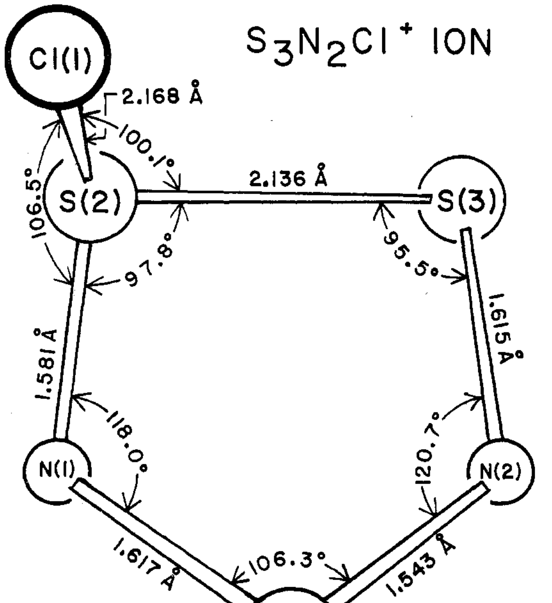


Figure 1.—Distances and angles in $S_3N_2Cl^+$. The estimated standard deviations are about 0.005 A on the distances and about 0.3° on the angles.

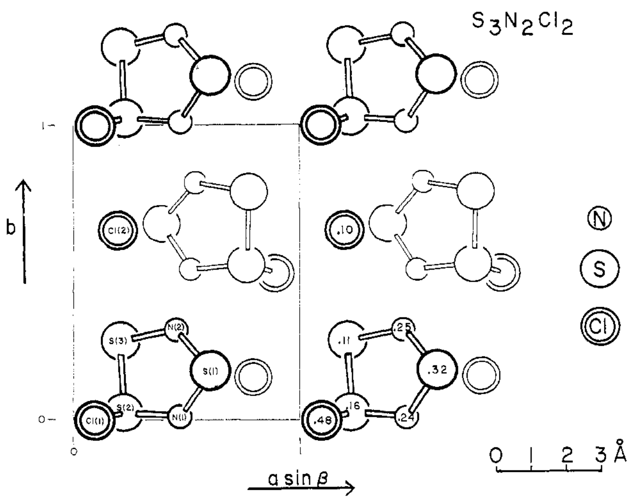
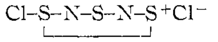
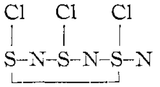
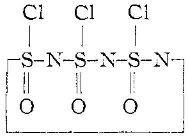
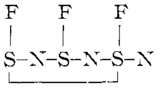
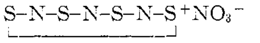


Figure 2.—Projection of $S_3N_2Cl_2$ down the *c* axis.

puckered five-membered ring with a Cl atom, Cl(1), attached to the thio sulfur, *i.e.*, Cl–S–N–S–N–S. The puckering can be described as a chair form in the following sense. If a plane is drawn through the three sulfur atoms, the Cl(1) atom is 2.09 A above the plane, N(1)

TABLE III
SELECTED BOND LENGTHS (A) IN SOME AZYL RING SYSTEMS

Formula	S–S	S–N	S–Cl	Ref
 $\mathrm{Cl{-}S{-}N{-}S{-}N{-}S^{+}Cl^{-}}$	2.136	1.543–1.617	2.168	This work
 $\mathrm{(NSCl)_3}$		1.600–1.614	2.084–2.150	10
 $\mathrm{(NSOCl)_3}$		1.546–1.578	1.991–1.999	9
 $\mathrm{(NSF)_3}$		1.540–1.660		11
 $\mathrm{S{-}N{-}S{-}N{-}S{-}N{-}S^{+}NO_3^{-}}$	2.064	1.493–1.582		12
$\mathrm{N_4S_4H_4}$		1.65		13

is 0.188 A below the plane, and the opposing N(2) atom is 0.140 A above the plane.

The five-membered ring in $S_3N_2Cl_2$ is the smallest such ring system in a series of cyclic azyl systems. Six-membered rings are present in $(NSOCl)_3$,[9] $(NSCl)_3$,[10] and $(NSF)_3$;[11] a seven-membered ring is present in $S_4N_3NO_3$;[12] and an eight-membered ring in $N_4S_4H_4$.[13] In all these cases the nitrogens are separated by sulfur, and halogen, when present, is bonded to sulfur. Table III shows a comparison of the common bonds in these compounds.

Figure 2 shows the packing in the crystal as seen in projection down the *c* axis.

Acknowledgment.—We thank Professor William Jolly, who first brought these substances to our attention, Dr. Norman Logan and Dr. Keith Maguire, who both stimulated our interest and prepared the material for the investigation, and Dr. Kenneth Wynne, who measured the density for our preliminary study of the crystal.

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(9) A. C. Hazell, G. A. Wiegers, and A. Vos, *Acta Cryst.*, **20**, 192 (1966).
(10) G. A. Wiegers and A. Vos, *ibid.*, **20**, 192 (1966).
(11) G. A. Wiegers and A. Vos, *ibid.*, **14**, 562 (1961).
(12) A. W. Cordes, R. F. Kruh, and E. K. Gordon, *Inorg. Chem.*, **4**, 681 (1965).
(13) E. W. Lund and S. R. Svendsen, *Acta Chem. Scand.*, **11**, 940 (1957).

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The Crystal Structure of Bis(ethylenediamine)palladium(II) Chloride

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The crystal structure of bis(ethylenediamine)palladium(II) chloride has been determined by three-dimensional X-ray diffraction techniques. The space group is $P\bar{1}$ with $a = 6.8607 \pm 0.0004$ A, $b = 8.3610 \pm 0.0008$ A, $c = 4.9940 \pm 0.0003$ A, $\alpha = 97.716 \pm 0.006°$, $\beta = 100.683 \pm 0.005°$, $\gamma = 108.571 \pm 0.006°$, and $Z = 1$. These cell parameters were determined by a least-squares refinement of thirty 2θ values measured on a single-crystal diffractometer. The errors indicated are the estimated standard deviations as obtained from this refinement and, as such, measure the precision of the determination and not necessarily the accuracy. The structure consists of discrete $[Pd(en)_2]^{2+}$ and Cl^- ions held together in layers parallel to (010) by a network of N–H···Cl hydrogen bonds. The N–Cl distances vary from 3.217 to 3.357 A. The palladium ion is four-coordinated by the nitrogen atoms of the ethylenediamine ligands in a planar configuration with an average Pd–N distance of 2.036 A.

Introduction

As part of a series of investigations carried out in this laboratory into the configuration of the ethylenediamine (en) ligand in coordination compounds we have determined the crystal structure of bis(ethylenediamine)palladium(II) chloride, $Pd[NH_2(CH_2)_2NH_2]_2Cl_2$. Cox and Preston[1] report that $Pd(en)_2Cl_2$ is isomorphous with $Pt(en)_2Cl_2$ and has almost identical cell dimensions. They report $Pt(en)_2Cl_2$ as crystallizing in space group $P\bar{1}$ and having lattice parameters as follows: $a = 8.37$ A, $b = 4.95$ A, $c = 6.86$ A, $\alpha = 100° 46'$, $\beta = 111° 40'$, $\gamma = 81° 56'$. When this cell is placed in the standardized form as recommended

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[1] E. G. Cox and G. H. Preston, *J. Chem. Soc.*, 1089 (1933).