

$x_{C1} = 0.318 \pm 0.001$, $y_{C1} = 0.278 \pm 0.001$, $z_{C1} = 0.084 \pm 0.001$,
 $x_C = 0.099 \pm 0.003$, $y_C = 0.003 \pm 0.005$, $z_C = 0.094 \pm 0.002$,

which are substantially unchanged from those previously reported (Reed & Lipscomb, 1953) except for the y and z parameters of the C atoms. Agreement factors of

$$\frac{\sum |F_o^2 - F_c^2|}{\sum F_o^2} = 0.17 \quad \text{and} \\ \frac{[\sum w(F_o^2 - F_c^2)^2]^{\frac{1}{2}}}{[\sum w F_o^4]^{\frac{1}{2}}} = 0.11$$

were obtained for all observed data, for our usual choice (Lavine & Lipscomb, 1954) of weights, w . The revised bond distances are C-Cl = 1.80 ± 0.02 , C-C = 1.55 ± 0.03 and Cl...Cl = 4.22 ± 0.01 Å. These values from the solid

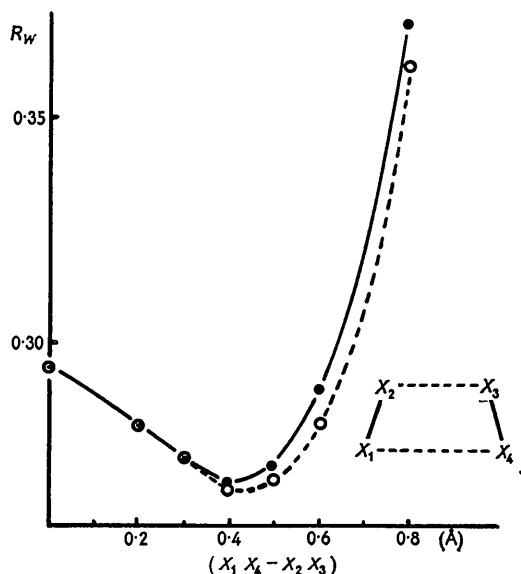


Fig. 1. Values of $R_w = \frac{\sum w(F_o^2 - F_c^2)^2}{\sum w F_o^4}$ for N_2O_2 disordered by a statistical center of symmetry². The average of X_1X_2 and X_3X_4 is 1.12 ± 0.02 Å, while the average of $X_2 \cdots X_3$ and $X_1 \cdots X_4$ is 2.40 ± 0.01 Å. The dotted line shows refinements for various $X_1X_2 - X_3X_4$ in which the shorter distance $X_2 \cdots X_3$ between NO groups is an N...N distance, while the solid curve indicates results for which an average scattering factor for N and O is employed for all atoms.

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Three thiourea complexes: A preliminary X-ray study. By M. PÉREZ RODRIGUEZ, M. CUBERO, R. VEGA, A. MORENTE and J. CASAS VAZQUEZ, *División de Ciencias Matemáticas, Médicas y de la Naturaleza, Universidad de Sevilla, Spain*

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A preliminary stage has been completed in the crystal structure determination of the following complexes:

(a) $Zn(NO_3)_2 \cdot Tu_4$, (b) $HgCl_2 \cdot Tu_3$, (c) $Ni(NO_3)_2 \cdot Tu_6$.

The last two have been obtained for the first time in our laboratory.

The dimensions of the unit cells are:

(a) $a = 22.53 \pm 0.06$, $b = 9.43 \pm 0.02$, $c = 8.90 \pm 0.03$ Å
 (space group D_{2h}^{10}).

phase are not in very good agreement with the values of C-Cl = 1.78 ± 0.01 and C-C = 1.49 ± 0.03 and Cl...Cl = 4.31 ± 0.02 Å obtained in an electron diffraction study (Ainsworth & Karle, 1952) of the gas, which contains about 27% of gauche molecules in addition to the trans-form to which these dimensions apply.

A reinvestigation of the N_2O_2 structure (Dulmage, Meyers & Lipscomb, 1953) has now led to definite conclusions concerning the molecular structure in the statistically disordered solid phase. A series of least-squares refinements with the assumption of isotropic temperature factors has been carried out for a set of models each of which averages to the statistical model previously described, and known with high precision. The results (Fig. 1) clearly favor the planar equilateral trapezoidal model, as compared with the rectangular model, and further suggest that the two NO groups are joined by an N...N bond about 2.18 Å in length. Estimates of errors of about ± 0.03 Å for this distance and the corresponding O...O distance of 2.62 Å are tied to the assumption of isotropic temperature factors, but the introduction of anisotropic temperature factors (which gave a singular matrix in our attempts to refine the structure) would probably require much larger estimates of these errors.

The origin of each of Figs. 1-6 of the N_2O_2 paper should be placed at the center of the figure rather than at the corner in order to agree with our atomic coordinates and the centrosymmetric equivalent positions x, y, z ; $\bar{x}, \bar{y}, \bar{z}$; $\frac{1}{2} - x, \frac{1}{2} + y, \bar{z}$; $\frac{1}{2} + x, \frac{1}{2} - y, z$ in $P2_1/a$. Also, in Fig. 2 the pair of numbers 0.621 and 0.601 should be interchanged, as should the pair 0.399 and 0.379.

We wish to thank Dr R. Dean Dragsdorf, who raised a question leading to our discovery of these mislabellings, for reading the N_2O_2 paper.

References

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$N = 4$ (calculated 4.05) for the number of formula units within the unit cell.

(b) $a = 8.65 \pm 0.03$, $b = 19.72 \pm 0.02$, $c = 8.07 \pm 0.02$ Å
 (space group C_{2h}^5);
 $\beta = 91.6 \pm 0.2^\circ$.

$N = 4$ (calculated 3.96).

(c) $a = 25.43 \pm 0.03$, $b = 9.22 \pm 0.03$, $c = 17.38 \pm 0.02$ Å;
 $\beta = 140.2 \pm 0.1^\circ$.

The possible space group being C_{2h}^6 and C_s^4 . $N = 4$ (calculated 3.96).