

**THE CRYSTAL STRUCTURE OF BISGLYCINE
CADMIUM CHLORIDE**

This is a preliminary note reporting the atomic coordinates in the crystal structure of bisglycine cadmium chloride.

Single crystals of bisglycine cadmium chloride $[(NH_2CH_2COOH)_2CdCl_2]$ were grown from a saturated aqueous solution containing glycine and cadmium chloride in 2 : 1 proportion. The crystal data are as follows: $a = 8.17$, $b = 8.91$, $c = 13.62 \text{ \AA}$, $\beta = 107^\circ$, $V = 948.14 \text{ \AA}^3$, F.W. = 333.42 g., $D_{meas} = 2.32 \text{ g. cm}^{-3}$, $D_{cal} = 2.33 \text{ g. cm}^{-3}$, $\mu(\text{CuK}\alpha) = 243.29 \text{ cm}^{-1}$, space group $P2_1/n$.

The three-dimensional intensity data (hkl , $K = 0$ to 8, $\mu r = 1.09$; hkL , $L = 0$ to 1, $\mu r = 1.31$) were collected using $\text{CuK}\alpha$ radiation by the multiple film equi-inclination Weissenberg technique. Spot-shape, absorption, Lorentz and polarisation corrections were applied. From an unsharpened three-dimensional Patterson synthesis, the heavy atoms—Cd, Cl (1) and Cl (2)—were located. Thereafter successive Fourier and difference Fourier syntheses revealed the rest of the structure. Least squares refinements was carried out using the LALS program (originally written by Gantzel, Sparks and Trueblood and modified later by Liminga of Upsala and also in the University of Madras) on an IBM 370/155 computer at the Indian Institute of Technology, Madras. With individual scale factors, the residual index at present is 0.124 for 1135 observed reflections. Further refinement is under progress. The present fractional atomic coordinates are presented in Table I.

TABLE I

Fractional atomic coordinates for bisglycine cadmium chloride

Atom	x	y	z
Cd	0.4738	0.0319	0.1299
Cl (1)	0.6852	0.1191	0.0343
Cl (2)	0.2767	-0.0412	0.2265
O (11)	0.6529	0.1582	0.2741
O (12)	0.8253	0.3493	0.3078
O (21)	0.3526	0.2588	0.0802
O (22)	0.2060	0.4546	0.0087
N (11)	0.8885	-0.0363	0.3614
N (22)	-0.0609	0.3066	-0.0872
C (11)	0.7956	0.2138	0.3022
C (12)	0.9481	0.1187	0.3442
C (21)	0.2162	0.3197	0.0294
C (22)	0.0698	0.2250	-0.0108

A view of the structure looking down the b -axis is shown in Fig. 1. The cadmium atom coordinates octahedrally with three chlorine atoms and three oxygen atoms form the glycines. The nitrogen atoms of glycines form three hydrogen bonds each, with carboxyl oxygens and a chlorine.

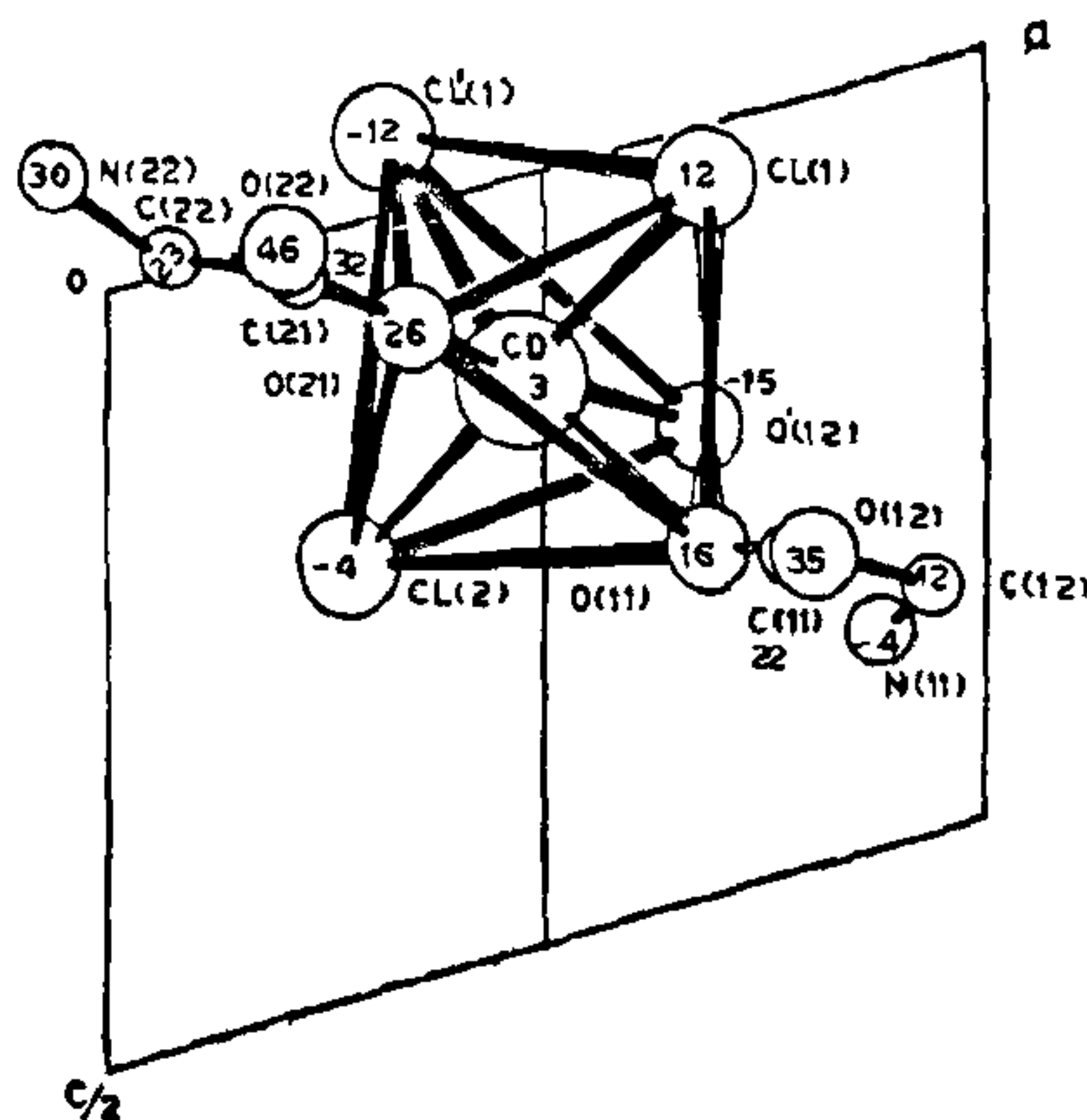


FIG. 1. The surroundings of cadmium in the asymmetric part of the unit cell. The two glycines in the asymmetric part are also shown.

Bisglycine cadmium bromide $[(NH_2CH_2COOH)_2CdBr_2]$ is isomorphous with bisglycine cadmium chloride with the following cell dimensions $a = 8.41$, $b = 9.01$, $c = 13.98 \text{ \AA}$, and $\beta = 108^\circ$. The R factor for the bromide compound is 0.107 for 1350 observed reflections.

The authors are thankful to Prof. K. S. Chandrasekaran and Prof. S. Balasubramanian for their interest in this work. One of the authors (RT) is thankful to the UGC for the award of a fellowship under the Faculty Improvement Programme.

School of Physics,
Madurai Kamaraj University,
Madurai 625 021,
October 12, 1979.

R. THULASIDHASS,
J. K. MOHANARAO.

**NITRATION OF AROMATIC COMPOUNDS
WITH AMMONIUM NITRATE IN
TRICHLOROACETIC ACID**

NITRATION of aromatic compounds using nitric acid in polyphosphoric acid has been reported earlier¹. Urea nitrate in polyphosphoric acid has been used as a nitrating agent for a number of aromatic compounds². The present paper describes nitration of aromatic compounds like naphthalene, *p*-nitrotoluene, benzaldehyde and phthalic acid with ammonium nitrate in trichloroacetic acid.

TABLE I

Nitration of Aromatic Compounds Using Ammonium nitrate in Trichloroacetic acid
($\text{NH}_4\text{NO}_3 = 0.05 \text{ mol}$; Organic Compound = 0.05 mol; Trichloroacetic acid = 0.05 mol)

Compound	Exptl. Condition	Product	Yield (%)
Naphthalene	At 35° for 1 hr	1-Nitronaphthalene ³	51
p-Nitrotoluene	At 60° for 1 hr	2,4-Dinitrotoluene ⁴	48
Benzaldehyde	At 100° for 1.5 hr	m-Nitrobenzaldehyde ⁶	48
Phthalic acid	At 100° for 2 hr	3-Nitrophthalic acid ⁵	42

By the above experimental conditions aromatic nitro compounds were obtained, which were identified (m.m.p., CO-TLC and IR) by comparison with authentic samples.

P. G. Chemistry Department, A. BALASUBRAMANIAN,
V.O. Chidambaram College,
Tuticorin 628 003,
January 8, 1980.

1. Kispersky, J. P. and Klager, J., *J. Am. Chem. Soc.*, 1955, **77**, 5433.
2. Nabar, V. B. and Kudav, N. A., *Indian J. Chem.*, 1977, **15B**, p. 89.
3. Vogel, A. I., *A Text Book of Practical Organic Chemistry*, (English Language Book Society and Longman Group Limited, London), Third Edition, 1975, p. 526.
4. —, *Ibid.*, 1975, p. 527.
5. —, *Ibid.*, 1975, p. 882.
6. —, *Ibid.*, 1975, p. 966.

SPECIFIC CHROMOGENIC REAGENT FOR THE IDENTIFICATION OF ENDRIN ON T.L.C.

A VARIETY of chromogenic spary reagents are in use for the detection of organo-chloro-insecticides on thin layer chromatography (TLC) plates. These reagents are not specific for endrin and give similar coloured spots with all chlorinated pesticides. Some workers have used rhodamine-B¹, fluorescent indicators² for the detection of organo-chloro-insecticides. In this paper stannous chloride in 50% hydrochloric acid followed by aqueous basic fuchsin dye solution is reported for the detection of endrin in formulations as well as in biological materials obtained in poisoning cases. This reagent is specific for endrin and sensitive at 5 μg concentration. Other organo-chloro-insecticides, carbamate insecticides and organo-phosphorous insecticides do not interfere with this test.

5 μl of each standard solution of endrin and other chlorinated pesticides in ethanol were spotted on the TLC plates coated with silica gel G, 0.25 mm thick. The chromatogram was developed with solvent mixture (1) hexane : acetone (9 : 1) and (2) hexane. The plate was air-dried and sprayed with stannous chloride reagent (5 gm stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) dissolved in 50 ml of concentrated hydrochloric acid and diluted to 100 ml with water) followed by 0.01% of aqueous basic fuchsin dye and was heated at 110° C for 5-10 minutes. Greenish-blue spot is observed for endrin.

Under identical conditions, representative samples of different dyes from the classified groups were tried but did not form the coloured complex with endrin. Only hydrochloric acid gave an intense coloured spot. The addition of stannous chloride to hydrochloric acid decolourised the background on TLC plate and increased the intensity of the coloured spot.

With this reagent all the organo-chloro-insecticides gave pink spots before heating. After heating the plate for about 10 minutes at 110° C the pink spot of endrin changed to greenish-blue colour, while the pink colour of isomers of endrin and other chlorinated pesticides disappeared completely.

Wiencke and Burke³ as well as Chau and Cochrane⁴ have shown that endrin forms endrin ketone and endrin aldehyde in presence of strong acids at 100° C. The well-known reaction of aldehyde and decolourised magenta³ forms the basis of the colour complex in this work. Stannous chloride in hydrochloric acid decolourises fuchsin and the endrin aldehyde formed restores the quinonoid structure of magenta and consequently forms bluish-green complex. Chlorides of metals such as Al^{+3} , Hg^{+1} , Hg^{+2} , Fe^{+3} , Ni^{+2} , Zn^{+2} , Sb^{+3} , Mg^{+2} were also found to give similar results.

The authors are thankful to Dr. B. N. Mattoo, Director, Forensic Science Laboratories, Maharashtra