Journal of Organometallic Chemistry, 52 (1973) 275–281

THE CRYSTAL STRUCTURE OF IODODIMETHYL(TRIMETHYLAMINE)-ALUMINUM

J. L. ATWOOD and P. A. MILTON
Department of Chemistry, University of Alabama, University, Alabama 35486 (U.S.A.)
(Received August 28th, 1972)

SUMMARY

The crystal structure of iododimethyl(trimethylamine)aluminum has been determined from single-crystal X-ray diffraction data collected by counter methods. $I(CH_3)_2Al\cdot N(CH_3)_3$ crystallizes in the orthorhombic space group *Pnma* with cell dimensions $a=12.59(1)$, $b=10.75(1)$, $c=7.58(1)$ Å, and $\rho_{calc}=1.58$ g·cm^{-3} for $Z=4$. Least-squares refinement gave a final weighted R value of 0.071 for 574 independent observed reflections. Each monomeric molecule lies on a crystallographic mirror plane with the trimethylamine and iododimethylaluminum groups in a staggered configuration about the aluminum–nitrogen bond.

INTRODUCTION

Adducts of the general formula $XR_2Al\cdot N(CH_3)_3$ may be prepared by two quite different methods. Smith and Wallbridge[1] have reported the straightforward addition of the components (eqn. 1) and we have discussed[2] the thermolysis of tetramethylammonium iodotrimethylaluminate. The latter represents to our knowledge the first reported instance of the *in situ* generation of trimethylamine from the tetramethylammonium cation in an organometallic compound. We wish to add further documentation of this behavior in the form of the completed crystal structure of iododimethyl(trimethylamine)aluminum.

$$Cl(CH_3)_2Al+N(CH_3)_3 \rightarrow Cl(CH_3)_2Al\cdot N(CH_3)_3 \qquad (1)$$

$$[N(CH_3)_4][Al(CH_3)_3)I] \xrightarrow{280°} I(CH_3)_2Al\cdot N(CH_3)_3+C_2H_6 \qquad (2)$$

EXPERIMENTAL

$I(CH_3)_2Al\cdot N(CH_3)_3$ was prepared by the thermolysis of $[N(CH_3)_4][Al(CH_3)_3I]$. Clear, colorless, rod-shaped, single crystals for the X-ray analysis were grown by vacuum sublimation and sealed in thin-walled glass capillaries.

Precession photographs showed that the crystals are orthorhombic with absences for $0kl$, $k+l=2n+1$, and for $hk0$, $h=2n+1$, so that the space group may be

TABLE 1

OBSERVED AND CALCULATED STRUCTURE FACTOR AMPLITUDES FOR IODODIMETHYL-(TRIMETHYLAMINE)ALUMINUM

H	K	OBS	CAL
******L= 0	*******		
0	2	219.1	266.0
0	4	115.5	168.7
0	6	131.9	139.0
0	8	84.6	80.5
0	10	42.6	43.0
0	12	25.2	24.1
2	0	127.1	159.3
2	1	26.5	12.9
2	2	159.8	158.2
2	3	30.6	31.7
2	4	88.6	104.8
2	5	0.0	9.2
2	6	48.8	50.2
2	7	0.0	8.3
2	8	46.8	40.9
2	9	0.0	5.3
2	10	28.9	22.2
2	11	0.0	6.2
4	0	88.8	63.8
4	1	144.4	142.2
4	2	31.2	34.0
4	3	102.2	109.8
4	4	13.8	12.7
4	5	82.3	84.0
4	6	11.1	11.6
4	7	52.1	49.7
4	8	0.0	5.7
4	9	23.1	25.2
4	10	0.0	3.2
4	11	15.9	13.2
6	0	19.5	13.9
6	1	109.7	119.9
6	2	13.3	13.2
6	3	128.8	120.4
6	4	0.0	7.1
6	5	81.7	82.2
6	6	0.0	2.7
6	7	45.6	49.6
6	8	0.0	3.6
6	9	34.7	31.7
8	0	26.2	25.8
8	1	59.6	60.7
8	2	18.3	21.2
8	3	45.8	49.9
8	4	17.5	17.3
8	5	39.4	38.4
8	6	12.1	13.0
8	7	23.2	23.1
8	9	0.0	11.7
10	0	38.3	41.9
10	1	14.5	19.1
10	2	39.8	39.8
10	3	14.3	17.7
10	4	33.5	32.4
10	5	11.1	12.7
10	6	20.6	23.0
10	7	0.0	7.0
10	8	17.8	18.3
12	0	30.9	29.9
12	1	0.0	6.1
12	2	24.0	27.5
12	3	0.0	5.5
12	4	18.5	22.3
12	5	0.0	4.5
12	6	17.9	16.2
14	0	6.4	8.9
14	1	0.0	3.8
******L= 1	*******		
0	1	75.8	94.0
0	3	86.8	103.4
0	5	64.1	72.9
0	7	41.1	39.7
0	9	17.1	19.4
8	0	52.9	50.3
8	1	23.3	22.8
8	2	43.5	45.0
8	3	19.3	18.4
8	4	32.0	31.8
8	5	0.0	12.3
8	6	21.0	24.2
8	7	0.0	7.8
8	8	16.5	15.1
8	9	0.0	4.9
9	0	15.3	18.9
9	1	0.0	6.3
9	2	20.2	19.5
9	3	0.0	4.2
9	4	17.1	15.8
9	5	0.0	3.1
9	6	0.0	9.2
9	7	0.0	2.6
9	8	0.0	5.7
9	9	0.0	2.7
10	0	19.9	18.8
10	1	30.9	31.9
10	2	15.8	16.9
10	3	30.2	28.2
10	4	0.0	12.9
10	5	22.7	20.5
10	6	0.0	8.8
10	7	14.0	13.2
10	8	0.0	5.4
11	0	31.2	30.9
11	1	0.0	5.6
11	2	29.0	28.8
11	3	0.0	5.4
11	4	0.0	21.8
11	5	0.0	1.7
11	6	0.0	17.8
11	7	0.0	2.6
12	0	0.0	2.9
12	1	22.2	21.8
12	2	0.0	2.4
12	3	19.3	19.0
12	4	0.0	2.2
12	5	0.0	14.4
12	6	0.0	1.6
13	0	8.6	8.5
13	1	0.0	4.0
13	2	10.2	7.8
13	3	0.0	1.7
13	4	0.0	6.2
14	1	0.0	10.4
******L= 2	*******		
0	0	34.7	34.5
0	2	40.2	45.4
0	4	20.5	25.6
0	6	0.0	8.6
0	8	10.8	7.5
0	10	0.0	3.3
1	0	8.4	20.1
1	1	171.1	171.2
1	2	25.1	24.5
1	3	95.7	114.5
1	4	28.0	26.4
1	5	72.1	82.9
1	6	12.9	11.5
1	7	55.2	54.6
1	8	10.4	8.1
1	9	31.2	29.8
1	10	0.0	5.3
1	11	15.7	14.3
2	0	130.5	98.4
2	1	52.8	47.9
2	2	50.7	55.1
2	3	33.0	37.1
2	4	21.0	32.4
10	2	0.0	2.2
10	3	17.0	17.7
10	4	0.0	2.0
10	5	14.6	14.6
10	6	0.0	1.5
10	7	10.6	10.8
11	0	7.1	6.3
11	1	24.2	23.8
11	2	0.0	6.1
11	3	19.5	20.4
11	4	0.0	5.1
11	5	14.8	14.9
11	6	0.0	3.3
11	7	8.7	9.6
12	0	0.0	2.9
12	1	0.0	1.4
12	2	0.0	2.8
12	3	0.0	3.1
13	0	0.0	5.0
13	1	17.1	17.4
13	2	0.0	4.8
13	3	13.8	15.1
******L= 3	*******		
0	1	31.5	34.5
0	3	0.0	17.3
0	5	11.0	15.5
0	7	14.1	12.6
0	9	0.0	6.1
1	0	84.2	85.7
1	1	79.1	69.7
1	2	81.0	91.9
1	3	32.1	45.4
1	4	76.8	78.0
1	5	38.5	41.7
1	6	49.1	46.7
1	7	33.6	30.0
1	8	24.2	28.6
1	9	14.4	16.5
1	10	11.9	11.2
1	11	10.4	10.2
2	0	9.6	17.0
2	1	44.0	39.4
2	2	22.2	21.1
2	3	31.3	31.4
2	4	15.3	16.8
2	5	18.0	21.0
2	6	0.0	8.2
2	7	0.0	15.2
2	8	0.0	5.6
2	9	0.0	9.5
2	10	0.0	3.1
2	11	0.0	5.4
3	0	119.0	107.6
3	1	98.2	94.5
3	2	100.3	99.8
3	3	84.6	81.6
3	4	82.6	86.0
3	5	52.7	54.9
3	6	62.5	61.7
3	7	39.4	31.9
3	8	33.8	35.7
3	9	29.2	24.6
3	10	17.6	20.1
3	11	14.9	12.9
4	0	0.0	9.1
4	1	16.1	11.4
4	2	0.0	6.9
4	3	4.8	7.1
4	4	0.0	4.2
4	5	0.0	4.3
4	6	0.0	2.4
4	7	0.0	4.9
4	8	0.0	1.6
4	9	0.0	2.4
1	4	0.0	5.3
1	5	41.4	45.5
1	6	0.0	3.1
1	7	13.6	31.7
1	8	0.0	1.4
1	9	23.6	19.0
1	10	0.0	0.9
2	0	70.2	61.0
2	1	44.6	41.4
2	2	52.2	55.0
2	3	49.5	54.7
2	4	38.4	42.6
2	5	41.7	41.9
2	6	33.4	30.1
2	7	31.3	30.0
2	8	18.4	17.2
2	9	19.0	14.0
2	10	10.3	8.8
3	0	26.5	11.1
3	1	54.1	46.7
3	2	38.2	32.5
3	3	26.8	31.8
3	4	11.0	26.8
3	5	19.4	22.1
3	6	17.5	15.1
3	7	18.3	15.4
3	8	0.0	7.9
3	9	0.0	7.4
3	10	0.0	4.1
4	0	51.5	52.0
4	1	62.6	60.6
4	2	56.3	51.3
4	3	57.9	53.8
4	4	44.4	42.7
4	5	34.9	39.0
4	6	26.7	24.2
4	7	23.6	25.0
4	8	14.6	19.9
4	9	16.4	14.4
4	10	13.1	11.0
5	0	47.4	49.4
5	1	17.8	15.7
5	2	51.3	49.1
5	3	16.3	13.0
5	4	44.5	41.0
5	5	0.0	4.8
5	6	24.1	26.9
5	7	0.0	3.9
5	8	18.4	16.7
6	0	0.0	1.3
6	1	15.1	10.8
6	2	0.0	1.6
6	3	21.0	24.5
6	4	0.0	1.5
6	5	20.2	19.7
6	6	0.0	0.9
6	7	8.4	10.6
6	8	0.0	0.6
7	0	15.7	37.0
7	1	0.0	1.8
7	2	16.4	14.4
7	3	0.0	1.0
7	4	31.8	30.2
7	5	0.0	9.5
7	6	21.0	20.7
7	7	0.0	4.4
7	8	10.5	11.7
8	0	31.7	31.9
8	1	28.4	31.4
8	2	18.3	29.8
8	3	31.2	24.1
8	4	24.0	24.1
8	5	19.6	21.0
8	6	17.0	18.0
6	4	39.8	42.3
6	5	0.0	1.3
6	6	31.1	30.3
6	7	0.0	1.1
6	8	22.4	19.5
7	0	13.2	15.2
7	1	0.0	7.0
7	2	14.6	14.9
7	3	0.0	5.1
7	4	13.5	13.2
7	5	0.0	4.0
7	6	0.0	10.1
8	0	28.6	28.0
8	1	13.6	11.5
8	2	28.7	26.0
8	3	13.7	12.0
8	4	22.5	20.9
8	5	10.2	8.5
8	6	14.2	14.5
9	0	0.0	2.3
9	1	0.0	1.8
9	2	0.0	1.8
9	3	0.0	2.0
9	4	0.0	1.6
10	0	11.4	11.8
10	1	22.2	22.6
10	2	10.7	11.0
10	3	14.9	14.7
******L= 6	*******		
0	0	79.8	81.1
0	2	41.7	55.8
0	4	38.0	44.2
0	6	31.7	31.8
0	8	21.4	20.3
1	0	0.0	1.7
1	1	20.1	27.1
1	2	0.0	1.6
1	3	21.6	25.1
1	4	0.0	1.5
1	5	20.4	18.9
1	6	0.0	1.3
1	7	11.6	12.1
1	8	0.0	1.1
2	0	46.4	42.5
2	1	15.6	14.2
2	2	39.7	34.9
2	3	14.4	12.0
2	4	29.3	30.1
2	5	14.5	4.7
2	6	23.4	20.6
2	7	0.0	4.8
2	8	16.0	12.9
3	0	22.1	20.2
3	1	0.0	5.5
3	2	18.8	18.1
3	3	0.0	4.2
3	4	0.0	16.9
3	5	0.0	1.5
3	6	0.0	10.1
3	7	0.0	1.6
4	0	26.0	16.7
4	1	19.5	18.9
4	2	15.8	14.8
4	3	11.9	11.0
4	4	0.0	10.6
4	5	28.5	25.0
4	6	0.0	0.5
4	7	14.8	10.6
5	0	31.7	27.8
5	1	0.0	1.6
5	2	28.0	25.7
5	3	11.3	4.1
5	4	18.4	21.1
5	5	0.0	1.6

0	10	0.0	0.0
0	11	4.6	8.1
1	0	124.5	121.4
1	1	15.1	9.1
1	2	95.8	108.4
1	3	38.3	27.8
1	4	85.2	88.9
1	5	0.0	1.6
1	6	66.1	62.0
1	7	9.2	7.4
1	8	34.7	35.2
1	9	0.0	1.8
1	10	18.1	19.5
1	11	0.0	7.4
2	0	121.2	110.7
2	1	94.4	105.8
2	2	96.9	92.2
2	3	120.3	113.6
2	4	61.6	67.4
2	5	73.0	79.2
2	6	47.1	47.2
2	7	40.4	42.0
2	8	30.1	28.4
2	9	21.6	22.1
2	10	16.9	15.7
2	11	12.7	10.7
3	0	12.4	22.2
3	1	65.8	56.6
3	2	55.2	44.1
3	3	10.4	20.1
3	4	34.6	36.6
3	5	23.5	21.8
3	6	0.0	13.1
3	7	20.1	17.0
3	8	0.0	7.0
3	9	0.0	5.1
3	10	0.0	2.5
3	11	0.0	1.9
4	0	128.1	117.1
4	1	61.8	64.5
4	2	92.1	92.4
4	3	68.8	64.9
4	4	61.8	67.0
4	5	52.2	49.9
4	6	53.5	57.5
4	7	29.8	29.2
4	8	33.6	31.6
4	9	12.5	15.8
4	10	17.8	15.6
4	11	0.0	8.7
5	0	13.9	10.0
5	1	91.7	90.9
5	2	0.0	6.2
5	3	81.3	79.8
5	4	0.0	4.2
5	5	57.4	58.0
5	6	0.0	4.6
5	7	38.7	39.0
5	8	0.0	2.0
5	9	27.8	24.6
5	10	0.0	2.6
5	11	14.8	13.4
6	0	95.8	91.3
6	1	8.3	6.7
6	2	80.8	80.2
6	3	10.7	6.7
6	4	54.5	60.3
6	5	0.0	9.9
6	6	44.1	42.8
6	7	0.0	5.7
6	8	28.9	26.1
6	9	0.0	2.1
6	10	15.6	12.8
7	0	0.0	5.7
7	1	59.1	57.6
7	2	13.6	11.9
7	3	47.5	48.1
7	4	16.5	12.6
7	5	33.2	34.5
7	6	0.0	8.3
7	7	20.3	23.9
7	8	0.0	7.9
7	9	16.2	14.1
7	10	0.0	5.9

2	5	31.9	31.0
2	6	36.1	34.8
2	7	23.5	20.7
2	8	22.2	19.5
2	9	9.7	12.4
2	10	9.0	9.4
2	11	9.0	7.5
3	0	105.5	95.6
3	1	101.4	95.9
3	2	93.3	92.6
3	3	74.8	74.7
3	4	67.2	71.8
3	5	42.2	50.1
3	6	47.4	45.3
3	7	32.4	12.7
3	8	26.5	26.4
3	9	24.7	20.2
3	10	12.9	13.6
3	11	10.4	9.8
4	0	51.4	49.1
4	1	0.0	10.6
4	2	54.2	49.7
4	3	10.2	21.9
4	4	35.4	36.2
4	5	0.0	7.7
4	6	18.1	22.9
4	7	0.0	2.4
4	8	20.4	18.0
4	9	0.0	2.1
4	10	14.1	11.2
5	0	90.2	96.8
5	1	44.7	40.2
5	2	91.3	90.9
5	3	20.5	27.0
5	4	76.2	71.5
5	5	14.4	17.7
5	6	46.2	47.5
5	7	14.1	12.8
5	8	25.3	26.0
5	9	0.0	7.4
5	10	15.5	11.8
6	0	12.3	10.8
6	1	15.9	16.5
6	2	11.4	12.3
6	3	8.9	10.4
6	4	10.2	14.3
6	5	9.4	8.6
6	6	12.4	10.7
6	7	0.0	1.4
6	8	0.0	6.6
6	9	0.0	2.2
6	10	0.0	5.3
7	0	60.7	66.5
7	1	25.8	24.9
7	2	66.3	65.1
7	3	20.1	20.4
7	4	56.1	51.4
7	5	18.6	17.2
7	6	33.7	34.5
7	7	18.6	12.4
7	8	19.6	20.1
7	9	0.0	6.4
7	10	11.1	10.9
8	0	18.9	20.9
8	1	30.1	35.5
8	2	22.6	20.4
8	3	41.0	35.0
8	4	19.7	17.9
8	5	26.5	25.3
8	6	10.9	14.3
8	7	12.7	16.1
8	8	9.9	9.8
8	9	11.8	11.1
9	0	38.6	42.6
9	1	32.4	32.8
9	2	37.6	39.1
9	3	30.4	27.9
9	4	31.7	32.1
9	5	22.7	21.5
9	6	22.1	23.2
9	7	15.5	14.1
9	8	10.3	13.8
10	0	0.0	2.3
10	1	19.2	20.4

4	10	0.0	1.7
5	0	21.1	24.2
5	1	82.3	71.1
5	2	17.2	37.4
5	3	48.5	52.0
5	4	34.8	30.7
5	5	34.4	38.2
5	6	17.2	20.6
5	7	27.5	25.1
5	8	10.2	13.1
5	9	15.0	11.7
5	10	0.0	7.5
6	0	13.0	17.5
6	1	19.6	18.4
6	2	20.8	18.8
6	3	20.9	14.7
6	4	17.6	15.6
6	5	13.9	11.8
6	6	0.0	8.2
6	7	0.0	9.0
6	8	0.0	4.9
6	9	0.0	6.2
7	0	23.5	28.9
7	1	67.3	61.0
7	2	10.5	26.5
7	3	48.6	50.1
7	4	24.2	21.5
7	5	33.2	36.9
7	6	11.6	11.7
7	7	24.4	24.2
7	8	12.7	11.0
7	9	15.2	13.5
8	0	20.9	17.5
8	1	13.1	11.6
8	2	18.3	16.6
8	3	8.8	9.1
8	4	14.5	11.6
8	5	10.6	8.2
8	6	0.0	4.8
8	7	11.3	5.1
8	8	8.8	6.8
9	0	15.0	11.4
9	1	42.3	42.3
9	2	29.3	28.5
9	3	35.4	16.7
9	4	22.1	22.6
9	5	23.6	27.5
9	6	17.7	15.7
9	7	18.4	19.5
9	8	12.1	9.6
10	0	0.0	1.8
10	1	0.0	5.6
10	2	0.0	1.7
10	3	0.0	5.1
10	4	0.0	1.8
10	5	0.0	3.5
10	6	0.0	3.5
10	7	0.0	1.7
11	0	17.3	18.1
11	1	0.0	10.1
11	2	17.6	17.0
11	3	0.0	8.4
11	4	15.6	13.5
11	5	0.0	6.3
11	6	9.2	8.6
12	0	0.0	4.3
12	1	0.0	4.1
12	2	0.0	3.9
12	3	0.0	3.3
12	4	0.0	3.2
13	0	15.1	15.0
13	1	0.0	6.9
13	2	13.7	14.0

********L= 4********

0	0	45.1	53.5
0	2	41.2	53.4
0	4	37.1	39.1
0	6	18.1	21.3
0	8	13.3	11.4
0	10	0.0	4.3
1	0	9.1	9.4
1	1	85.5	82.4
1	2	0.0	8.1
1	3	55.1	63.7

8	7	9.6	11.9
8	8	13.7	12.4
9	0	9.2	17.5
9	1	11.1	11.2
9	2	15.4	16.9
9	3	12.3	11.4
9	4	17.2	11.9
9	5	0.0	9.9
9	6	9.3	8.8
10	0	14.5	16.9
10	1	16.1	15.5
10	2	16.0	11.6
10	3	0.0	11.4
10	4	13.4	10.6
10	5	10.7	10.4
10	6	8.6	7.2
11	0	0.0	9.1
11	1	15.0	15.5
11	2	10.7	8.6
11	3	12.4	11.4
11	4	0.0	7.1
11	5	11.2	10.5
12	0	11.3	10.4
12	1	0.0	6.1
12	2	11.9	9.7

********L= 5********

0	1	67.1	49.1
0	3	66.2	79.1
0	5	58.6	60.1
0	7	19.7	19.6
0	9	25.8	23.8
1	0	12.8	16.0
1	1	13.9	14.5
1	2	13.5	18.5
1	3	14.4	12.9
1	4	21.7	19.1
1	5	0.0	11.0
1	6	16.4	12.0
1	7	0.0	8.0
1	8	0.0	7.4
1	9	0.0	5.5
2	0	33.0	27.2
2	1	73.4	65.6
2	2	23.1	23.6
2	3	52.1	56.9
2	4	11.3	17.4
2	5	44.6	43.3
2	6	13.9	11.7
2	7	33.8	28.2
2	8	0.0	6.2
2	9	15.7	15.8
3	0	17.6	10.8
3	1	0.0	0.9
3	2	0.0	7.9
3	3	0.0	1.6
3	4	0.0	5.6
3	5	0.0	1.4
3	6	0.0	6.0
3	7	0.0	0.5
3	8	0.0	5.5
3	9	0.0	1.0
4	0	54.7	54.6
4	1	23.5	22.0
4	2	55.5	50.8
4	3	21.3	19.0
4	4	40.5	40.0
4	5	12.8	14.5
4	6	25.6	27.4
4	7	0.0	8.4
4	8	20.8	16.9
4	9	0.0	1.9
5	0	0.0	6.3
5	1	22.8	19.8
5	2	0.0	5.8
5	3	16.2	16.9
5	4	0.0	5.8
5	5	0.0	14.2
5	6	0.0	4.3
5	7	11.0	11.1
5	8	0.0	1.7
6	0	65.2	57.9
6	1	0.0	1.9
6	2	55.0	53.2
6	3	0.0	1.8

5	6	15.0	16.1
6	0	7.0	7.8
6	1	19.9	19.7
6	2	0.0	7.2
6	3	14.5	15.5
6	4	0.0	4.9
6	5	24.1	27.6
7	0	14.0	10.5
7	1	0.0	3.1
7	2	0.0	4.4
7	3	0.0	2.5
7	4	0.0	7.1
7	5	0.0	1.4
8	0	0.0	2.1
8	1	16.6	14.1
8	2	0.0	2.1
8	3	11.0	14.1
8	4	0.0	1.6
8	5	13.8	11.6
9	0	0.0	3.5
9	1	12.6	11.4
9	2	0.0	1.4
10	0	13.9	14.6

********L= 7********

0	1	11.5	15.1
0	3	11.6	11.4
0	5	14.3	10.7
1	0	41.0	40.8
1	1	0.0	3.1
1	2	24.8	17.7
1	3	0.0	2.5
1	4	24.0	23.0
1	5	0.0	1.5
1	6	21.5	21.1
2	0	0.0	10.2
2	1	16.9	16.5
2	2	0.0	9.3
2	3	17.9	15.1
2	4	0.0	7.6
2	5	13.0	11.7
2	6	0.0	5.7
3	0	18.4	16.0
3	1	15.0	15.7
3	2	14.2	14.5
3	3	15.7	11.1
3	4	0.0	11.2
3	5	0.0	9.1
3	6	0.0	7.7
4	0	11.7	7.7
4	1	0.0	14.3
4	2	0.0	8.6
4	3	14.0	13.0
4	4	0.0	4.6
4	5	0.0	10.4
5	0	0.0	1.3
5	1	27.0	30.8
5	2	0.0	1.3
5	3	26.5	26.9
5	4	0.0	1.4
5	5	20.1	20.5
6	0	0.0	8.0
6	1	0.0	1.5
6	2	0.0	7.2
6	3	0.0	1.2
6	4	0.0	5.4
7	0	0.0	2.0
7	1	21.1	21.4
7	2	0.0	1.8
7	3	18.4	18.9
8	0	10.4	13.3

********L= 8********

0	0	0.0	10.1
0	4	0.0	9.2
1	0	0.0	11.8
1	1	16.2	20.0
1	2	0.0	11.0
1	3	16.3	17.2
2	0	0.0	0.5
2	1	0.0	4.0
2	2	0.0	0.7
2	3	0.0	4.1
3	0	18.2	19.1
3	1	19.1	21.7
3	2	15.1	18.0
3	3	17.6	19.3

either *Pnma* or $Pna2_1$. The lattice parameters ($T=24°$, $\lambda=1.5418$ Å) determined from a least-squares refinement of $(\sin\theta/\lambda)^2$ values for 10 reflections are: $a=12.59(1)$, $b=10.75(1)$, $c=7.58(1)$ Å, and $\rho_{\mathrm{calc}}=1.58$ g·cm^{-3} for $Z=4$.

Data were taken on an ENRAF-NONIUS CAD-4 diffractometer with Ni-filtered copper radiation. The crystal, a rod of dimensions $0.15\times0.15\times0.65$ mm, was aligned so that the rod axis (c^*) was coincident with the φ axis of the goniometer. The diffracted intensities were collected by the $\omega-2\theta$ scan technique with a take-off angle of 1.5°. The scan rate was variable and was determined by a fast 20° min^{-1} prescan. If more than 35 net counts were found, a slow scan was carried out; if not, the reflection was considered to be unobserved. Calculated speeds for the slow scan based on the net intensity gathered in the prescan ranged from 7° min^{-1} to 0.8° min^{-1}. Background counts were collected for 25% of the total scan time at each end of the scan range. For each intensity the scan width was determined by the equation:

$$\text{scan range} = A + B\tan\theta$$

where $A=1.0°$ and $B=0.5°$. Aperture settings were determined in a like manner with $A=4$ and $B=4$ mm. The crystal-to-source and crystal-to-detector distances were 21.6 and 20.8 cm, respectively. The lower level and upper level discriminators of the pulse height analyzer were set to obtain a 95% window centered on the Cu-K_α peak. Two standard peaks were measured periodically during data collection; no significant variation in the intensities was noticed.

The standard deviations of the intensities, σ_I, were estimated from the formula

$$\sigma = \{[C_N + (T_C/2T_B)^2(B_1+B_2)] + k^2[C_N + (T_C/2T_B)^2(B_1+B_2)]^2\}^{\frac{1}{2}}$$

where C_N is the counts collected during scan time T_C, B_1 and B_2 are background intensities, each collected during the background time T_B, and $k=0.03$. One unique octant of data was measured out to $2\theta=130°$. A total of 720 reflections were collected of which 574 had intensities greater than their estimated standard deviations. The intensities were corrected in the usual manner for Lorentz, polarization, and absorption[3] ($\mu=252$ cm^{-1}). The calculated transmission factors ranged from 0.028 to 0.075.

Fourier calculations were made with the ALFF[4] program. The full-matrix, least-squares refinement was carried out using the Busing and Levy program ORFLS[5]. The function $\Sigma\, W(|F_o|-|F_c|)^2$ was minimized. No correction was made for extinction. Neutral atom scattering factors were taken from the compilation of Cromer and Waber[6], that for iodine having been corrected for anomalous dispersion[7]. Final bond distances were computed with the aid of the ORFFE program[8]. Crystal structure illustrations were obtained with the program ORTEP[9].

STRUCTURE DETERMINATION AND REFINEMENT

The iodine atom was located on the initial three-dimensional Patterson map, and Fourier maps phased on the iodine position revealed all the nonhydrogen atoms in the asymmetric unit. Subsequent isotropic refinement in the centric space group *Pnma* let to an $R_1=\Sigma(|F_o|-|F_c|)/(\Sigma F_o)=0.112$. Anisotropic refinement with unit weights further lowered R_1 to 0.081, and $R_2=[\Sigma\, W(|F_o|-|F_c|)^2/\Sigma(F_o)^2]^{\frac{1}{2}}\times 100=0.089$. A check of $W(|F_o|-|F_c|)^2$ *vs.* $(\sin\theta/\lambda)$ and F_o showed no irregularities with the exception of the very intense reflections. These were subsequently given reduced weights and several cycles of least-squares refinement led to final values of $R_1 = 0.079$ and $R_2 = 0.071$. Unobserved reflections and three reflections (200, 020, 040) which appeared to suffer from secondary extinction were not included. The largest parameter shifts in the final cycle of refinement were less than 0.07 of their estimated standard deviations. A final difference Fourier map showed one peak of approximately 0.7 $e^-/\text{Å}^3$ close to the iodine atom position, and several of the same magnitude in the vicinity of expected hydrogen atom positions; however, no effort was made to locate or include hydrogen atom coordinates. Refinement was attempted in the acentric space group $Pna2_1$, but high parameter correlations and essentially no improvement in the reliability index demonstrated that the correct choice was *Pnma*. The final value of the standard deviation of an observation of unit weight was 1.92. Final observed and calculated structure factor amplitudes are listed in Table 1. The final values of the positional and thermal parameters are given in Table 2.

TABLE 2A

FINAL ATOMIC POSITIONAL PARAMETERS[a]

Atom	*x/a*	*y/b*	*z/c*
I	−0.0408(1)	0.2500	0.1540(2)
Al	0.1370(5)	0.2500	−0.0153(7)
N	0.0884(13)	0.2500	−0.2685(21)
C(1)	0.2107(12)	0.0931(14)	0.0246(23)
C(2)	0.1874(18)	0.2500	−0.3871(28)
C(3)	0.0243(14)	0.1369(16)	−0.3136(17)

[a] Standard deviations in parentheses refer to last digit quoted.

TABLE 2B

ANISOTROPIC TEMPERATURE FACTORS ($\times 10^4$)[a]

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
I	103(1)	153(2)	221(3)		22(2)	
Al	73(4)	65(5)	173(11)		−27(6)	
N	91(13)	63(15)	231(34)		22(18)	
C(1)	99(13)	82(15)	375(41)	6(13)	−48(19)	19(22)
C(2)	106(19)	141(28)	232(51)		66(25)	
C(3)	133(15)	132(18)	212(29)	−66(14)	−13(18)	−32(19)

[a] Anisotropic thermal parameters defined by $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$

DISCUSSION

The structure (Fig. 1) is such that in the space group *Pnma*, the aluminum, nitrogen, iodine, and one of the nitrogen-bonded methyl-carbon atoms lie on a crystallographic mirror plane. The configuration of the trimethylamine and iododimethylaluminum groups about the aluminum–nitrogen bond is staggered. This has also proven to be the preferred geometry in the two related compounds, $Cl_3Al\cdot N$-

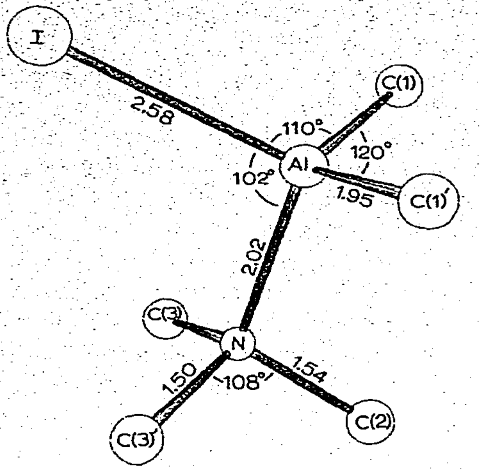

Fig. 1. Molecular structure of iododimethyl(trimethylamine)aluminum.

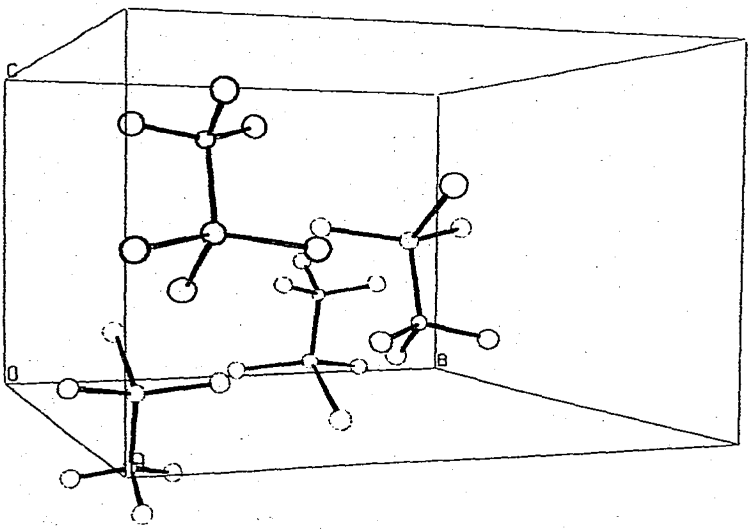

Fig. 2. Packing in one unit cell of iododimethyl(trimethylamine)aluminum.

$(CH_3)_3$[11] and $(CH_3)_3Al \cdot NC_7H_{13}$[12]. The aluminum–nitrogen bond length of 2.02 Å falls within the extreme values of 1.96Å[11] and 2.06Å[12] reported for other organoaluminum donor–acceptor complexes. The aluminum–carbon is shorter (1.95Å) than one normally finds for the situation in which the hydrogen atoms on a methyl group have not been located[13]. However, this may be attributed to the enhanced positive charge on the aluminum atom because of the presence of the electronegative iodine atom. By the same token, the carbon–aluminum–carbon bond angle of 120° is due to the second-order rehybridization of the aluminum atom demanded by the halide. The aluminum–iodine distance of 2.58Å seems reasonable, although there are none with which to compare. The packing in the unit cell is illustrated by Fig. 2. The only intramolecular contacts of less than 4.0Å are between iodine and methyl groups (3.97(2)Å) and methyl groups (3.98(2)Å).

ACKNOWLEDGEMENTS

We are grateful to the National Science Foundation for support of this work under Grant GP-24852, and for departmental assistance in the purchase of the X-ray diffractometer. The authors also thank Mr. W. J. Cook for aid in obtaining the crystal structure illustrations.

REFERENCES

1 C. A. Smith and M. G. H. Wallbridge, *J. Chem. Soc. A*, (1967) 7.
2 J. L. Atwood and P. A. Milton, *J. Organometal. Chem.*, 36 (1972) C1.
3 D. J. Wehe, W. R. Busing and H. A. Levy, *ORABS, A Fortran Program for Calculating Single-Crystal Absorption Corrections, Report ORNL-TM*-229, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1962.
4 C. R. Hubbard, C. O. Quicksall and R. A. Jacobson, *USAEC Reports IS*-2625, Ames Laboratory, Iowa State University, Ames, Iowa, 1971.
5 W. R. Busing, K. O. Martin and H. A. Levy, *ORFLS, A Fortran Crystallographic Least-Squares Program, Report ORNL-TM*-305, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1962.
6 D. T. Cromer and J. T. Waber, *Acta Crystallogr.*, 18 (1965) 104.
7 D. T. Cromer, *Acta Crystallogr.*, 18 (1965) 17.
8 W. R. Busing, K. O. Martin and H. A. Levy, *ORFFE, A Fortran Crystallographic Function and Error Program, Report ORNL-TM*-306, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1964.
9 C. K. Johnson, *ORTEP, A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations, Report ORNL*-3794, Revised, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1965, p. 70.
10 D. F. Grant, R. C. G. Killean and J. L. Lawrence, *Acta Crystallogr., Sect. B*, 25 (1969) 377.
11 C. D. Whitt, L. M. Parker and J. L. Atwood, *J. Organometal. Chem.*, 32 (1971) 291.
12 J. L. Atwood and G. D. Stucky, *J. Organometal. Chem.*, 13 (1968) 53.