The only literature data, which are directly comparable with the present results, are those of Kehiaian et al. (*10*) for 2,5,8-trioxanonane–*n*-decane. Near $x = 0.5$, these are about 3% higher than our curve.

Excess enthalpies at 304.15 K have been reported for binary mixtures of 2,5,8-trioxanonane with *n*-decane and with *n*-dodecane (*11*). For equimolar mixtures, these are ~250 J mol^{-1} higher than our results at 298.15 K. In combination with our results, they indicate unrealistically large positive molar excess isobaric heat capacities (~40 J K^{-1} mol^{-1}), which are in sharp contrast to the small negative values obtained from direct measurements of the heat capacities of mixtures of the same ether with *n*-heptane (*6*).

Registry No. 2,5,8-Trioxanonane, 111-96-6; 2,5,8,11,14-pentaoxapentadecane, 143-24-8; *n*-hexane, 110-54-3; *n*-decane, 124-18-5; *n*-dodecane, 112-40-3; *n*-heptane, 142-82-5.

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Vapor Pressures of Ammonium Bromide + Ammonia and Ammonium Iodide + Ammonia Solutions

Hideki Yamamoto, Seiji Sanga, and Junji Tokunaga*

Department of Chemical Engineering, Faculty of Engineering, Kansai University, Yamate-cho, Suita, Osaka, 564 Japan

Vapor pressures for ammonium bromide + ammonia and ammonium iodide + ammonia solutions were measured in the concentration ranges of 10–65 wt % and 10–75 wt % at various temperatures, respectively. The observed vapor pressures were fitted to Antoine's equation, using the method of least squares. Furthermore, Antoine's constants for both solutions were expressed as fourth-degree functions of concentration of ammonium halides. In addition, vapor pressures for saturated ammonia solutions of ammonium halide were measured over the temperature range of 0–80 °C. The calculated vapor pressures by these equations at 25 °C were compared with Hunt's data, and good agreement was obtained. Vapor pressure measurements were carried out by using a strain gauge transducer (up to 2.0 MPa), and the accuracy of this measurement was ±0.001 MPa. The vapor pressures calculated by Antoine's equation agreed with the observed values within a deviation of ±2.7%.

Introduction

Reaction products from ammonium halides and liquid ammonia are referred as ammine complexes or ammoniated salts. Recently, these complexes in the liquid phase have been utilized as a working system for chemical energy storage and chemical heat pumps (*1*, *2*, *3*).

Table I. Vapor Pressure of Saturated Ammonia Solution of Ammonium Halide

temp, °C	press., MPa	temp, °C	press., MPa
	NH_4Br-NH_3 System		
0.5	0.094	47.3	0.456
5.8	0.115	50.0	0.491
10.6	0.138	54.5	0.564
16.0	0.162	59.1	0.647
20.3	0.190	63.8	0.740
25.5	0.231	69.0	0.855
30.9	0.267	73.5	0.967
34.3	0.294	78.6	1.098
41.6	0.374	83.2	1.255
	NH_4I-NH_3 System		
0.6	0.043	44.6	0.182
8.5	0.053	48.3	0.198
14.5	0.066	50.4	0.212
16.0	0.074	53.8	0.226
19.1	0.080	59.0	0.259
25.0	0.101	60.1	0.261
28.4	0.110	63.8	0.286
35.8	0.138	68.9	0.315
39.6	0.158	82.6	0.469

Ammonium bromide + ammonia and ammonium iodide + ammonia solutions were valuable as the working system (*4*). However, the physical properties of these systems, especially the observed values for vapor pressure, were not reported sufficiently. Vapor pressures of these two systems at 25 °C

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Table II. Vapor Pressure for the NH_4Br-NH_3 System

		observed values			
temp, °C	concn, wt %	press., MPa	temp, °C	concn, wt %	press., MPa
0.2	10.20	0.420	0.2	19.80	0.411
8.9	10.24	0.574	8.8	19.83	0.559
10.0	10.25	0.596	10.2	19.84	0.587
15.9	10.29	0.727	16.0	19.86	0.713
20.1	10.32	0.833	20.2	19.90	0.817
25.0	10.35	0.972	25.1	19.93	0.952
30.1	10.38	1.133	29.9	19.96	1.100
35.2	10.41	1.315	35.3	19.99	1.287
39.8	10.44	1.496	39.9	20.01	1.464
44.8	10.49	1.714	44.9	20.04	1.677
0.2	31.56	0.407	0.5	36.50	0.391
8.7	31.59	0.551	8.7	36.53	0.522
10.0	31.60	0.576	10.2	36.54	0.555
15.9	31.63	0.701	15.9	36.57	0.665
20.1	31.65	0.802	20.7	36.60	0.779
25.0	31.68	0.934	25.1	36.63	0.896
29.8	31.70	1.079	30.2	36.67	1.053
35.4	31.74	1.268	35.1	36.71	1.209
39.7	31.76	1.429	37.4	36.73	1.288
44.9	31.78	1.645	44.8	36.77	1.595
0.2	41.99	0.361	0.2	44.84	0.352
8.4	42.04	0.490	8.7	44.90	0.476
9.6	42.05	0.513	9.8	44.91	0.486
15.2	42.09	0.623	16.0	44.97	0.605
20.6	42.14	0.739	20.6	45.03	0.705
24.3	42.19	0.845	25.0	45.08	0.816
29.8	42.25	0.985	30.2	45.15	0.945
34.6	42.31	1.137	35.2	45.23	1.100
38.7	42.37	1.277	39.9	45.31	1.257
43.0	42.42	1.432	45.0	45.33	1.443
50.0	42.48	1.711	50.0	45.38	1.648
0.2	48.82	0.313	0.6	51.08	0.294
8.5	48.99	0.432	8.8	51.16	0.401
10.1	48.91	0.541	9.9	51.17	0.419
15.7	48.97	0.551	15.6	51.24	0.513
20.1	49.03	0.637	20.3	51.30	0.606
25.0	49.10	0.749	25.5	51.38	0.711
29.8	49.18	0.874	30.1	51.47	0.825
34.4	49.27	1.005	34.6	51.56	0.949
39.9	49.37	1.160	39.8	51.67	1.099
43.6	49.46	1.292	44.8	51.81	1.280
49.8	49.53	1.507	50.0	51.95	1.424
0.4	53.30	0.271	0.2	55.35	0.238
8.6	53.39	0.369	8.3	55.43	0.327
9.9	53.40	0.389	9.6	55.45	0.343
15.7	53.47	0.472	15.6	55.52	0.427
20.4	53.55	0.557	20.3	55.59	0.504
25.2	53.63	0.654	25.1	55.67	0.594
29.9	53.72	0.760	29.3	55.75	0.681
34.9	53.83	0.888	34.2	55.86	0.793
40.0	53.96	1.030	39.1	55.98	0.920
44.8	54.10	1.183	44.2	56.12	1.049
50.1	54.25	1.329	49.7	56.28	1.231
53.0	54.38	1.438	52.1	56.33	1.314
0.3	58.19	0.203	0.4	62.22	0.156
8.4	58.27	0.284	8.7	62.30	0.223
9.6	58.29	0.297	10.6	62.32	0.240
15.2	58.35	0.366	15.3	62.39	0.290
20.4	59.43	0.442	20.9	62.46	0.349
25.2	58.51	0.521	25.3	62.54	0.413
30.1	58.60	0.608	29.9	62.63	0.485
34.9	58.70	0.708	34.8	62.74	0.573
39.6	58.82	0.820	39.8	62.86	0.672
44.7	58.96	0.950	44.8	63.01	0.771
49.7	59.12	1.102	49.9	63.17	0.889
53.9	59.27	1.245	54.5	63.33	1.017
0.4	65.05	0.118			
8.1	65.11	0.167			
9.4	65.22	0.177			
15.1	65.28	0.220			
20.7	65.34	0.272			
25.4	65.42	0.326			
29.8	65.48	0.382			
34.9	65.58	0.454			
39.6	65.69	0.536			
44.8	65.82	0.631			
50.0	65.96	0.740			
54.4	66.10	0.843			
58.8	66.26	0.956			
63.0	66.44	1.069			
68.5	66.65	1.234			

were reported in the concentration range from 3.2 wt % to saturated solution by Hunt and Larsen (*5*). The vapor pressures over a wide temperature range and at various concentrations have no been reported yet.

In this present work, vapor pressures for ammonium bromide + ammonia and ammonium iodide + ammonia solutions were determined in the concentration ranges 10-65 wt % and 10-75 wt % at various temperatures, respectively. The constants *A* and *B* for Antoine's equation were determined from the observed data. Furthermore, these constants are expressed as a fourth-degree function of concentration by using the method of least squares. In addition, the vapor pressures for saturated ammonia solution of ammonium halide were measured at 0-80 °C.

This paper deals with the measurement of vapor pressure for ammonium bromide + ammonia and ammonium iodide + ammonia solutions.

Experimental Section

***Materials*.** Ammonium bromide (NH_4Br) and ammonium iodide (NH_4I) from Wako Pure Chemicals Industries Co., Ltd. were of guaranteed reagent grade and were specified as the pure grade having minimum purities of 99.5% and used without further purification. The powdered crystal was thoroughly dried at 100 °C and stored over silica gel in a desiccator.

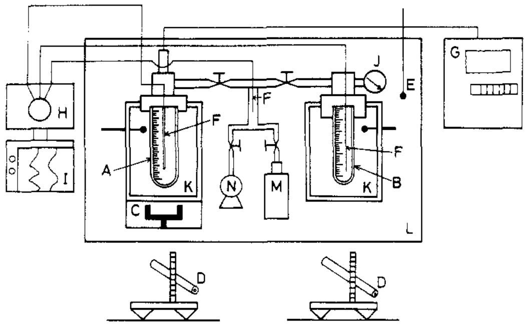

Figure 1. Schematic diagram of the experimental apparatus: A, reaction vessel; B, ammonia vessel; C, magnetic stirrer; D, cathetometer; E, thermocouple; G, digital pressure gauge; H, selective switch, I, recorder; J, Bourdon gauge; K, constant temperature water bath; L, constant temperature air bath; M, ammonia cylinder; N, vacuum pump.

Ammonia gas of 99.99% purity was provided by Seitetu Kagaku Co. Ltd.

***Experimental Apparatus*.** The schematic diagram of the experimental apparatus is shown in Figure 1 (*6*). Two pres-

Table III. Vapor Pressures for the NH_4I-NH_3 System

		observed values			
temp, °C	concn, wt %	press., MPa	temp, °C	concn, wt %	press., MPa
0.2	10.18	0.422	0.4	20.85	0.414
8.6	10.21	0.573	8.9	20.87	0.562
9.9	10.22	0.599	10.1	20.88	0.585
15.9	10.25	0.735	15.8	21.03	0.708
20.2	10.29	0.846	20.2	21.07	0.811
25.0	10.31	0.984	25.1	21.11	0.950
29.9	10.34	1.143	29.9	21.13	1.098
34.7	10.37	1.316	34.9	21.16	1.269
39.9	10.40	1.526	40.1	21.20	1.468
45.0	10.44	1.754	45.2	21.26	1.676
0.1	31.45	0.407	0.1	45.03	0.359
9.0	31.47	0.560	8.4	45.06	0.484
10.1	31.48	0.582	9.8	45.07	0.509
15.7	31.53	0.702	15.6	45.10	0.615
20.2	31.57	0.811	20.3	45.13	0.718
25.0	31.60	0.942	24.9	45.16	0.830
29.8	31.63	1.088	29.6	45.19	0.944
35.1	31.67	1.269	34.8	45.24	1.112
40.2	31.69	1.463	39.9	45.28	1.271
45.0	31.72	1.665	45.1	45.34	1.463
0.6	50.63	0.320	0.5	54.89	0.281
8.8	50.65	0.435	8.8	54.94	0.383
10.3	50.66	0.457	10.1	54.95	0.399
15.8	50.70	0.553	15.9	54.99	0.489
20.5	50.74	0.645	20.9	55.04	0.576
25.1	50.78	0.748	25.0	55.09	0.661
29.7	50.83	0.859	29.5	55.15	0.761
35.1	50.89	1.013	35.0	55.22	0.891
39.8	50.96	1.164	39.9	55.30	1.032
43.2	51.00	1.277	44.8	55.38	1.188
50.0	51.05	1.561	47.5	55.43	1.277
0.3	57.64	0.248	0.6	62.04	0.194
8.8	57.69	0.339	8.8	62.11	0.267
10.1	57.71	0.361	10.3	62.14	0.282
15.7	57.76	0.440	15.6	62.16	0.342
20.7	57.81	0.519	20.4	62.20	0.404
24.8	57.85	0.597	24.9	62.25	0.471
29.6	57.91	0.688	29.5	62.31	0.546
34.8	57.98	0.808	34.8	62.39	0.646
39.9	58.06	0.931	40.9	62.47	0.758
44.8	58.15	1.070	44.9	62.56	0.877
50.3	58.27	1.255	50.7	62.69	1.029
51.4	58.28	1.289	55.5	62.79	1.176
55.1	58.35	1.402	58.9	62.88	1.295
0.6	64.89	0.158	0.3	67.47	0.121
8.8	64.94	0.220	8.6	67.52	0.174
10.0	64.95	0.231	10.1	67.53	0.182
15.5	64.99	0.284	15.6	67.57	0.227
20.9	65.04	0.340	20.6	67.61	0.271
25.1	65.09	0.396	25.0	67.65	0.317
29.8	65.14	0.460	29.6	67.70	0.372
35.4	65.22	0.551	35.0	67.77	0.446
40.4	65.30	0.641	40.1	67.85	0.524
45.0	65.38	0.739	44.9	67.92	0.609
50.4	65.49	0.861	50.8	68.03	0.724
55.6	65.61	1.000	55.7	68.13	0.834
59.1	65.69	1.067	59.3	68.22	0.922
63.7	65.81	1.234	63.5	68.33	1.036
65.9	65.86	1.301	68.8	68.45	1.175
71.5	65.94	1.497	72.5	68.56	1.290
0.6	70.89	0.086	0.3	73.40	0.064
8.9	70.93	0.124	8.5	73.43	0.092
10.3	70.94	0.132	10.3	73.44	0.099
15.9	70.98	0.164	15.6	73.47	0.124
20.8	71.01	0.199	20.4	73.50	0.151
25.1	71.05	0.235	24.7	73.54	0.179
29.7	71.10	0.277	29.8	73.57	0.214
34.9	71.19	0.331	35.1	73.62	0.258
40.3	71.32	0.397	40.0	73.68	0.307
45.8	71.39	0.467	45.1	73.75	0.366
50.9	71.49	0.557	50.7	73.83	0.439
55.7	71.58	0.646	55.7	73.91	0.512
63.9	71.65	0.817	59.3	73.98	0.571
68.8	71.68	0.933	63.9	74.06	0.648
72.9	71.75	1.042	69.2	74.18	0.756
77.7	71.86	1.175	73.8	74.29	0.847
81.4	71.96	1.290	78.3	74.40	0.952
83.6	72.03	1.350	83.4	74.56	1.091
0.6	75.89	0.046			
8.6	75.92	0.066			
10.1	75.93	0.070			
15.8	75.95	0.090			
20.3	75.97	0.108			
25.4	76.00	0.134			
29.4	76.03	0.157			
34.5	76.07	0.192			
39.5	76.12	0.228			
44.4	76.17	0.271			
50.0	76.24	0.328			
55.1	76.31	0.388			
58.6	76.37	0.433			
63.7	76.45	0.500			
68.6	76.54	0.576			
73.3	76.64	0.653			
77.4	76.73	0.732			
82.9	76.87	0.850			

Table IV. Dependence of Antoine's Constants on Solute Concentration in the Unsaturated Ammonia Solution of Ammonium Halide

	NH_4Br-NH_3 system		NH_4I-NH_3 system	
const	A	B	A	B
X^4	$a_4 = 2.32324 \times 10^{-8}$	$b_4 = 3.94255 \times 10^{-5}$	$a_4 = -9.97644 \times 10^{-9}$	$b_4 = 1.90584 \times 10^{-5}$
X^3	$a_3 = 2.83431 \times 10^{-6}$	$b_3 = -2.23307 \times 10^{-3}$	$a_3 = 5.87745 \times 10^{-6}$	$b_3 = -1.12902 \times 10^{-4}$
X^2	$a_2 = -2.39924 \times 10^{-4}$	$b_2 = 3.72501 \times 10^{-2}$	$a_2 = -3.98604 \times 10^{-4}$	$b_2 = -5.96942 \times 10^{-2}$
X^1	$a_1 = 2.77311 \times 10^{-3}$	$b_1 = -3.43111 \times 10^{-1}$	$a_1 = 4.87144 \times 10^{-3}$	$b_1 = 1.09151$
X^0	$a_0 = 9.62756$	$b_0 = 994.38$	$a_0 = 9.65725$	$b_0 = 998.79$
ϵ^a	±2.7		±2.6	

[a] Pressure deviation = $100(P_{\text{obsd}} - P_{\text{cald}})/P_{\text{obsd}}$, %.

sure-resistant glass vessels (A and B) were used in this experiment. One is the 100-mL reaction vessel (A) for measuring the vapor pressure of ammonia solution and the other is 20-mL ammonia vessel (B) for measuring the amount of liquid ammonia. The volumes of these two vessels were measured by using a cathetometer (D) with an accuracy of 0.02% of full volume. During the experiment, ammonia solution was agitated by a magnetic stirrer (C). The two glass vessels were immersed in two constant temperature water bathes. The temperature control of each bath was maintained by using a refrigeration unit combined with a electric relay unit and thermoregulator. The accuracy of this water bath was better than

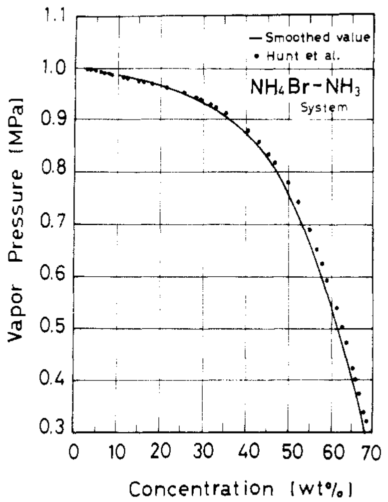

Figure 2. Comparison of smoothed vapor pressure calculated by Antoine's equation with Hunt's data at 25 °C.

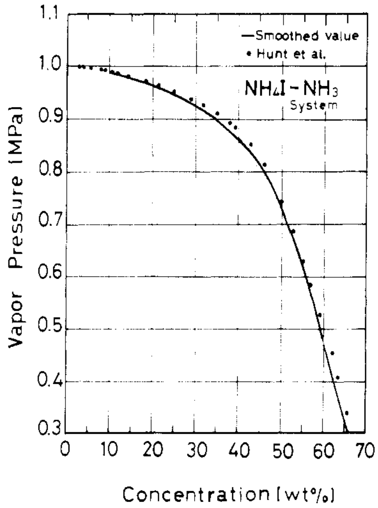

Figure 3. Comparison of smoothed vapor pressure calculated by Antoine's equation with Hunt's data at 25 °C.

±0.05 °C. Furthermore, these two vessels were covered with a constant temperature air bath (L), and it was kept at 25 ±1.0 °C during the experiment. The temperature of ammonia solutions was measured with alumel–chlomel thermocouples, calibrated by a standard mercury thermometer, and its accuracy was estimated ±0.05 °C. The vapor pressure was measured with a strain gauge transducer (G) with an accuracy of ±0.001 MPa. The concentration of ammonia solution was determined from the volume change of liquid ammonia and mass of solute (ammonium halides) at various conditions.

Experimental Method. Weighed solid ammonium halides (NH_4Br or NH_4I) are charged in the reaction vessel (A). The pipe arrangement and the vessels shown in Figure 1 are kept at 80 °C under vacuum for 2 h in order to remove moisture from the system. Then, these vessels are cooled down to room temperature. By chilling the ammonia vessel (B), liquid ammonia is allowed to be introduced into its vessel from the cylinder (M). The amount of liquid ammonia was determined from the difference of liquid level in the ammonia vessel. The correction of concentration for the liquid ammonia phase corresponding to temperature change was carried out by using the equation of state for ammonia gas (*7*) in each experiment.

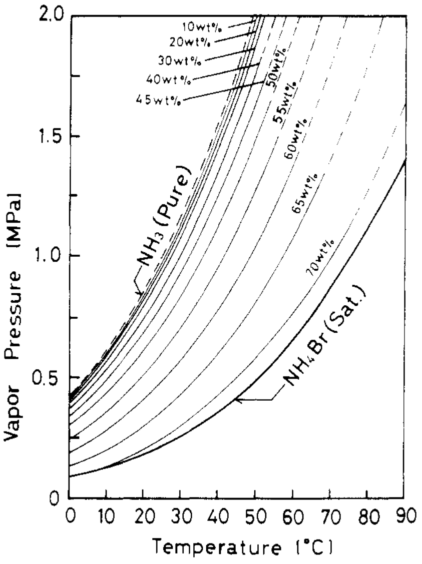

Figure 4. Smoothed vapor pressure for the NH_4Br–NH_3 system.

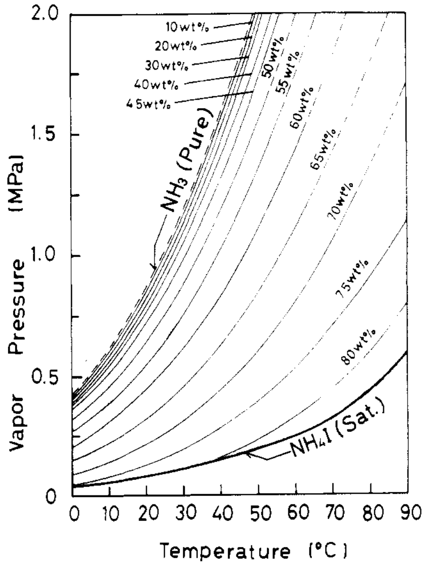

Figure 5. Smoothed vapor pressure for the NH_4I–NH_3 system.

Results and Discussion

Vapor Pressure Measurement for Saturated Ammonia Solution of Ammonium Halide. Table I shows the observed vapor pressures for saturated ammonium bromide + ammonia and ammonium iodide + ammonia solutions. The observed values for ammonium bromide + ammonia solutions have good agreement with Hunt's data (*4*). However, for ammonium iodide + ammonia solutions, the maximum deviation from Hunt's data was 1.9% in this experiment.

Vapor Pressure Measurement for Unsaturated Ammonia Solution of Ammonium Halide. Tables II and III show observed vapor pressures for ammonium bromide + ammonia and ammonium iodide + ammonia solutions. The comparison of smoothed vapor pressure for these systems at 25 °C with Hunt's data (4) is presented in Figures 2 and 3. These values show a lower value than Hunt's data, but a good agreement at low concentration range was obtained. Furthermore, the following form of Antoine's equation was applied to experimental data in the concentration range from 10 wt % to saturated solution. The Antoine constants (*A* and *B*) for each system were expressed as the fourth-degree function of ammonium halide concentration and these constants were are in Table IV.

$$\log P = A - B/(C + t) \qquad (1)$$

$$A = a_0 + a_1X + a_2X^2 + a_3X^3 + a_4X^4 \qquad (2)$$

$$B = b_0 + b_1X + b_2X^2 + b_3X^3 + b_4X^4 \qquad (3)$$

$$C = 247.885 \qquad (4)$$

Figures 4 and 5 show the smoothed vapor pressures calculated by using these equations. These smoothed vapor pressures agreed with observed ones with an accuracy of ±2.7% in the measured concentration range.

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Glossary

a_i	constants for eq 3
A	constant for Antoine's equation
b_i	constants for eq 4
B	constant for Antoine's equation
C	constant for Antoine's equation
P	vapor pressure, Pa
t	temperature, °C
X	weight percent concentration of ammonium halide

Greek Letter

ϵ	deviation, %

Registry No. NH_3, 7664-41-7; NH_4Br, 12124-97-9; NH_4I, 12027-06-4.

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NEW COMPOUNDS

Synthesis and Insecticidal Activity of Some 2,5-(Fluoroalkoxyphenyl)-1,3,4-oxadiazoles and Their *N*,*N'*-Dibenzoylhydrazine Precursors

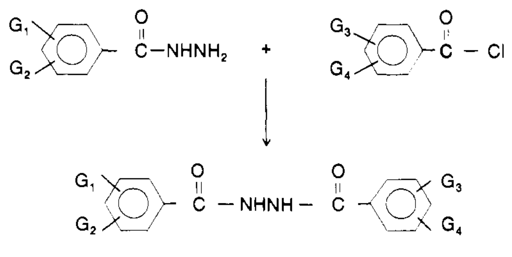
John P. Idoux* and Kathleen S. Gibbs-Rein
Department of Chemistry, Lamar University, Beaumont, Texas 77710

John T. Gupton and Glenn N. Cunningham
Department of Chemistry, University of Central Florida, Orlando, Florida 32816

The synthesis and IR/NMR characterization of 10 2,5-(fluoroalkoxyphenyl)-1,3,4-oxadiazoles and 15 fluoroalkoxydiacylhydrazines are described. Five of the oxadiazoles and 11 of the diacylhydrazines have been screened for insecticidal activity against *Drosophila melanogaster*.

In view of the biological activity properties and the variety of other important diverse uses of many organofluoro materials (*1*, *2*) and because of our continuing interest (*3*, *4*) in the synthetic methodology of such materials, we report here the syntheses of some 2,5-(fluoroalkoxyphenyl)-1,3,4-oxadiazoles (I) and of their *N*,*N'*-dibenzoylhydrazine precursors (II). A preliminary investigation of the insecticidal activity of these compounds is also reported.

Scheme I



IIa-i

G_1 = H or Haloalkoxy
G_2 = Haloalkoxy
G_3 = 2-Cl
G_4 = 4-Cl

Unsymmetrical *N*,*N'*-dibenzoylhydrazines (IIa-i, G_1 = H or haloalkoxy, G_2 = haloalkoxy; G_3 = 2-Cl, G_4 = 4-Cl; Scheme I) were prepared by reaction of the corresponding substituted benzhydrazide with 2,4-dichlorobenzoyl chloride. The required benzhydrazides were obtained by reaction of hydrazine hydrate with the appropriately substituted methyl benzoates as described previously by Smith (*5*) and the requisite methyl fluo-