

were expressed as the fourth-degree function of ammonium halide concentration and these constants were in Table IV.

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

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

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

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

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 $\pm 2.7\%$ in the measured concentration range.

Acknowledgment

We express our gratitude to Mr. A. Shimomura for his experimental work and to Mr. A. Kurata for his useful suggestions.

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

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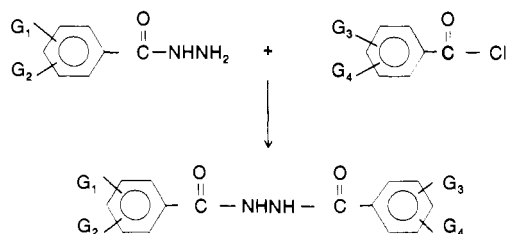
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The synthesis and IR/NMR characterization of 10 2,5-(fluoroalkoxyphenyl)-1,3,4-oxadiazoles and 15 fluoroalkoxydiacylhydrazines are described. Five of the oxadiazoles and 11 of the diacylhydrazines have been screened for insecticidal activity against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

Unsymmetrical N,N' -dibenzoylhydrazines (IIa-i, $G_1 = \text{H}$ or haloalkoxy, $G_2 = \text{haloalkoxy}$; $G_3 = 2\text{-Cl}$, $G_4 = 4\text{-Cl}$; 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

Scheme I



IIa-i

$G_1 = \text{H}$ or Haloalkoxy

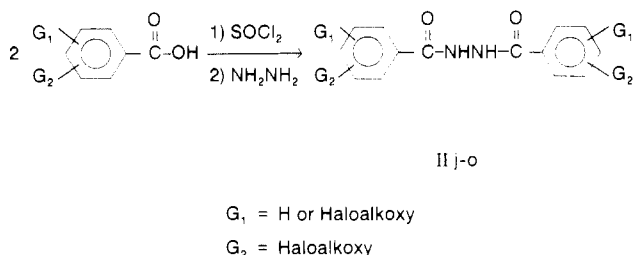
$G_2 = \text{Haloalkoxy}$

$G_3 = 2\text{-Cl}$

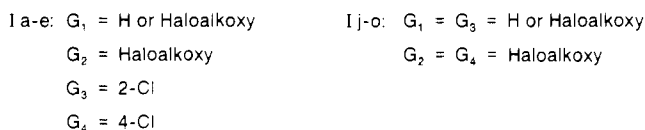
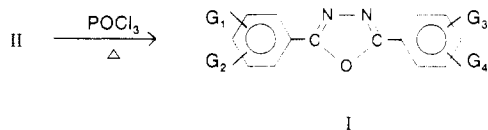
$G_4 = 4\text{-Cl}$

with the appropriately substituted methyl benzoates as described previously by Smith (5) and the requisite methyl fluo-

Scheme II



Scheme III



roalkoxybenzoates were prepared as reported by Alperman and von Werner (6). The symmetrical *N,N'*-dibenzoylhydrazines (IIj-o, G₁ = H or haloalkoxy and G₂ = haloalkoxy; Scheme II) were prepared by reaction of 2 equiv of the appropriately substituted benzoyl chloride with hydrazine. The 2,5-disubstituted-1,3,4-oxadiazoles (I) were prepared (Scheme III) by cyclodehydration of the corresponding *N,N'*-dibenzoylhydrazine (II) in refluxing phosphorous oxychloride (7, 8).

The structures of the compounds were confirmed by IR and NMR analyses. All compounds reported here were found to be greater than 90% pure by TLC. As reported previously (9) for other benzhydrazides, the benzhydrazides prepared for this study were readily soluble in 5% HCl, whereas the subsequent *N,N'*-dibenzoylhydrazines and oxadiazoles are insoluble. All of the hydrazines and oxadiazoles gave NMR and IR spectra characteristic of the substituent hydrohaloalkoxy groups. For example, chemical shifts of ~6–7 ppm, expected splitting patterns (*i.e.*, triplet of triplets for the H of HCF₂CF₂, doublet of triplets for the H of HCClCF₂, triplet for the H of HCCl₂CF₂), vicinal coupling constants of ~3–4 Hz, and geminal coupling constants of ~44–54 Hz for the hydrogen of the hydrohaloalkoxy groups of all substituted dibenzoylhydrazines and oxadiazoles were in accord with the values assigned previously by other workers (6, 10). In addition, all IR spectra contained strong, broad absorption at ~1100–1300 cm⁻¹ characteristic of C–F stretching (11). Other characteristic features in the IR and NMR of all dibenzoylhydrazines and oxadiazoles included the following. For the *dibenzoylhydrazines*, strong secondary amide stretch at ~3200–3300 cm⁻¹ [primary amine stretch noted at ~3400 cm⁻¹ in the precursor benzhydrazides was absent], a single, strong carbonyl absorption at ~1610–1640 cm⁻¹ [a second strong absorption, noted by us and by previous workers (9, 12, 13) in the same region in the precursor benzhydrazide, was absent], strong C–H bending absorptions at ~810–870, cm⁻¹ due to meta- and para-disubstituted benzene rings [also observed in the precursor benzhydrazides] and absence of a broad singlet at 3.6–4.0 ppm in the NMR [observed in the precursor benzhydrazides due to the primary amine hydrogens in CONHNH₂]. For the *oxadiazoles*, absence of secondary amide and carbonyl stretching absorptions, the appearance of oxadiazole ring absorptions at ~970 and 1020–1030 cm⁻¹ due to C–O and at ~1590 to 1610 cm⁻¹ due

to C=N (14, 15) and strong C–H bending at ~810–870 cm⁻¹ due to meta- and para-disubstituted benzene rings.

Eleven of the *N,N'*-dibenzoylhydrazines and five of the oxadiazoles were screened for insecticidal activity at a 100 ppm level against *Drosophila melanogaster*. With one exception (compound II d), the *N,N'*-dibenzoylhydrazines have low to moderate activity at a 100 ppm level. However, four of the five oxadiazoles (compounds Ia–d) had a 100% total kill rate at the 100 ppm level.

Experimental Section

All melting points are uncorrected and were recorded on a Thomas-Hoover capillary melting point apparatus. IR spectra were recorded as Nujol mulls on either a Perkin Elmer Model 1420 infrared spectrometer or a Nicolet MXS FT-IR. The NMR spectra were obtained in solution (see particular compound for solvent) at 60 MHz with a Varian EM-360 spectrometer with (CH₃)₄Si as internal standard.

General Procedure for Preparation of Unsymmetrical *N,N'*-Dibenzoylhydrazines (IIa–I, G₁ = H or Haloalkoxy, G₂ = Haloalkoxy; G₃ = 2-Cl, G₄ = 4-Cl; Scheme I). The appropriate substituted benzoic acid hydrazide (0.05 mol), sodium carbonate (0.05 mol), 60 mL of tetrahydrofuran, and 60 mL of water were added in a 250-mL, three-necked, round-bottomed flask which was equipped with a 125-mL addition funnel, reflux condenser, thermometer, and stirring bar. After dropwise addition (30 min) of 2,4-dichlorobenzoyl chloride (0.05 mol), the reaction mixture was stirred at room temperature for 1 h. The solid collected after removal (rotary evaporator) of the solvent was washed with water, dried at reduced pressure (Kugelrohr, 1 mm), and recrystallized.

The following compounds are reported in Table I: *N*-[4-(1,1,2,2-tetrafluoroethoxy)benzoyl]-*N'*-(2,4-dichlorobenzoyl)hydrazine (IIa); *N*-[4-(1,1,2-trifluoro-2-chloroethoxy)benzoyl]-*N'*-(2,4-dichlorobenzoyl)hydrazine (IIb); *N*-[4-(1,1-difluoro-2,2-dichloroethoxy)benzoyl]-*N'*-(2,4-dichlorobenzoyl)hydrazine (IIc); *N*-[3-(1,1,2,2-tetrafluoroethoxy)benzoyl]-*N'*-(2,4-dichlorobenzoyl)hydrazine (II d); *N*-[3-(1,1,2-trifluoro-2-chloroethoxy)benzoyl]-*N'*-(2,4-dichlorobenzoyl)hydrazine (II e); *N*-[3-(1,1-difluoro-2,2-dichloroethoxy)benzoyl]-*N'*-(2,4-dichlorobenzoyl)hydrazine (II f); *N*-[3,5-bis(1,1,2,2-tetrafluoroethoxy)benzoyl]-*N'*-(2,4-dichlorobenzoyl)hydrazine (II g); *N*-3,5-bis(1,1,2-trifluoro-2-chloroethoxy)benzoyl]-*N'*-(2,4-dichlorobenzoyl)hydrazine (II h); *N*-[3,5-bis(1,1-difluoro-2,2-dichloroethoxy)benzoyl]-*N'*-(2,4-dichlorobenzoyl)hydrazine (II i).

General Procedure for Preparation of Symmetrical *N,N'*-Dibenzoylhydrazines (IIj-o, G₁ = H or Haloalkoxy and G₂ = Haloalkoxy; Scheme II). The appropriate substituted benzoic acid (0.0163 mol) and 75 mL of toluene were added in a 250-mL, three-necked, round-bottomed flask equipped with a reflux condenser and a 125-mL addition funnel. Thionyl chloride (0.0498 mol) was then added dropwise and the solution was refluxed for 72 h. After cooling to room temperature, the solvent was removed (rotary evaporator) and the IR of the residue was recorded. If O–H absorption was observed, additional thionyl chloride and solvent were added and the solution was refluxed for 24 h. The resulting benzoyl chloride was added to 75 mL of tetrahydrofuran followed by dropwise addition of aqueous sodium bicarbonate (0.0161 mol) and hydrazine (0.008 mol). After stirring for 1 h at room temperature, the solvent was removed (rotary evaporator). The resulting solid was collected, dried at reduced pressure (Kugelrohr, 1 mm), and recrystallized.

The following compounds are reported in Table II: *N,N'*-bis[3,3'-(1,1,2,2-tetrafluoroethoxy)benzoyl]hydrazine (IIj); *N,N'*-bis[3,3'-(1,1,2-trifluoro-2-chloroethoxy)benzoyl]hydrazine (IIk); *N,N'*-bis[3,3'-(1,1-difluoro-2,2-dichloroethoxy)benzoyl]hydrazine (IIl); *N,N'*-bis[4,4'-(1,1,2-trifluoro-2-chloroethoxy)-

Table I. Properties of *unsym-N,N'*-Dibenzoylhydrazines (IIa-i)

| compd. no. | yield, % | mp, °C | IR ^a ν, cm ⁻¹ | NMR ^b δ (J, Hz) |
|------------|----------|---------|---|---|
| IIa | 92 | 198–200 | 3220, 1640, 1460, 1220, 1110, 870 | 6.35 (tt, $J_v = 4$, $J_g = 56$, 1 H), 7.16–7.65 (m, 5 H), 8.14 (d, $J = 10$, 2 H) |
| IIb | 78 | 198–200 | 3220, 1640, 1180, 1090, 1020, 850 | 7.10 (dt, $J_v = 4$, $J_g = 48$, 1 H), 7.33–7.65 (m, 5 H), 8.20 (d, $J = 8$, 2 H) |
| IIc | 84 | 210–212 | 3220, 1645, 1250, 1170, 1020, 890 | 7.47–8.00 (m, 6 H), 8.40 (d, $J = 8$, 2 H) |
| IIId | 94 | 165–168 | 3220, 1650, 1470, 1300, 1200, 1130, 1005, 870 | 6.48 (tt, $J_v = 4$, $J_g = 52$, 1 H), 7.58 (d, $J = 2$, 3 H), 7.63 (s, 2 H), 8.00 (d, $J = 2$, 2 H) |
| IIe | 96 | 169–171 | 3240, 1645, 1470, 1300, 1200, 1090, 1005, 880 | 6.66 (dt, $J_v = 4$, $J_g = 50$, 1 H), 7.45 (d, $J = 2$, 3 H), 7.59 (s, 2 H), 7.98 (t, $J = 2$, 2 H) |
| IIIf | 74 | 171–172 | 3300, 1640, 1590, 1275, 1120, 1000, 830 | 6.36 (t, $J = 4$, 1 H), 7.33 (d, $J = 2$, 3 H), 7.68 (s, 2 H), 7.93 (d, $J = 2$, 2 H) |
| IIg | 93 | 157–161 | 3210, 1600, 1470, 1200, 1130, 1010, 880 | 6.90 (tt, $J_v = 4$, $J_g = 52$, 2 H), 7.48 (t, $J = 2$, 1 H), 7.69 (s, 2 H), 7.78 (s, 1 H), 8.00 (d, $J = 2$, 2 H) |
| IIh | 93 | 110–114 | 3200, 1600, 1470, 1170, 1100, 1005, 870 | 7.20 (dt, $J_v = 4$, $J_g = 44$, 2 H), 7.28 (s, 1 H), 7.60 (m, 3 H), 7.90 (s, 2 H) |
| IIi | 97 | 110–112 | 3200, 1600, 1470, 1160, 1100, 1000, 830 | 7.15 (t, $J = 4$, 2 H), 7.45 (t, $J = 2$, 1 H), 7.65 (s, 3 H), 7.93 (d, $J = 2$, 2 H) |

^aRecorded as Nujol mulls. All indicated absorptions were of strong to moderately strong intensity. ^bRecorded in 10% DMSO-*d*₆/CDCl₃ solution. J_v = vicinal coupling constant, J_g = geminal coupling constant, s = singlet, d = doublet, t = triplet, dt = doublet of triplets, and tt = triplet of triplets.

Table II. Properties of *sym-N,N'*-Dibenzoylhydrazines (IIj-o)

| compd no. | yield, % | mp, °C | IR ^a ν, cm ⁻¹ | NMR ^b δ (J, Hz) |
|-----------|----------|---------|---|---|
| IIj | 85 | 154–156 | 3220, 1610, 1580, 1210, 1110, 1000, 810 | 6.34 (tt, $J_v = 4$, $J_g = 54$, 2 H), 7.32–7.58 (m, 4 H), 7.74–8.08 (m, 4 H) |
| IIk | 82 | 116–120 | 3210, 1610, 1580, 1200, 1090, 1000, 810 | 7.08 (dt, $J_v = 4$, $J_g = 48$, 2 H), 7.40–7.63 (7, 4 H), 7.90–8.10 (m, 4 H) |
| IIl | 80 | 96–100 | 3300, 1640, 1580, 1310, 1170, 1070, 1000, 820 | 6.48 (t, $J = 4$, 2 H), 7.65 (s, 4 H), 8.16 (s, 4 H) |
| IIIm | 98 | 155–158 | 3240, 1600, 1650, 1270, 1170, 1080, 1010, 810 | 6.90 (dt, $J_v = 4$, $J_g = 46$, 2 H), 7.35 (d, $J = 8$, 4 H), 8.18 (d, $J = 8$, 4 H) |
| IIIn | 86 | 140–144 | 3210, 1630, 1580, 1300, 1200, 1130, 1000, 870 | 6.64 (tt, $J_v = 4$, $J_g = 54$, 4 H), 7.48 (s, 2 H), 8.09 (s, 4 H) |
| IIo | 90 | 209–212 | 3210, 1630, 1590, 1270, 1170, 1100, 1000, 870 | 6.66 (dt, $J_v = 4$, $J_g = 48$, 4 H), 7.50 (s, 2 H), 8.18 (s, 4 H) |

^aSee footnote a, Table I. ^bSee footnote b, Table I.

Table III. Properties of 2,5-Disubstituted-1,3,4-oxadiazoles (I)

| compd no. | yield, % | mp, °C | IR ^a ν, cm ⁻¹ | NMR ^b δ (J, Hz) |
|-----------|----------|---------|---|--|
| Ia | 90 | 137–141 | 1590, 1040, 1010, 970 | 6.34 (tt, $J_v = 3$, $J_g = 54$, 1 H), 7.50 (d, $J = 10$, 2 H), 7.44–7.70 (m, 2 H), 8.20 (d, $J = 8$, 1 H), 8.29 (d, $J = 10$, 2 H) |
| Ib | 89 | 135–139 | 1590, 1040, 1020, 970, 870 | 6.69 (dt, $J_v = 3$, $J_g = 48$, 1 H), 7.46 7.40–7.68 (m, 2 H), 8.13 (d, $J = 8$, 1 H) 8.20 (d, $J = 8$, 2 H) |
| Ic | 93 | 91–94 | 1595, 1045, 1020, 970, 830 | 6.15 (t, $J = 3$, 1 H), 7.78 (d, $J = 8$, 2 H), 7.97 (s, 2 H), 8.30 (s, 1 H), 8.62 (d, $J = 3$, 1 H) |
| Id | 90 | 69–71 | 1595, 1050, 1005, 930, 840 | 6.44 (tt, $J_v = 3$, $J_g = 54$, 1 H), 7.45–7.70 (m, 4 H), 7.98–8.21 (m, 3 H) |
| Ie | 88 | 71–73 | 1595, 1050, 1020, 970, 850 | 7.30 (dt, $J_v = 3$, $J_g = 46$, 1 H), 7.55–7.84 (m, 4 H), 8.00–8.34 (m, 3 H) |
| Ij | 80 | 148–151 | 1595, 1550, 1490, 1300, 1190, 1100, 1000, 870 | 6.04 (tt, $J_v = 4$, $J_g = 54$, 2 H), 7.83 (d, $J = 4$, 4 H), 8.21 (s, broad, 4 H) |
| II | 76 | 129–131 | 1590, 1550, 1380, 1260, 1150, 1080, 1000, 810 | 5.43 (t, $J = 4$, 2 H), 7.11 (m, 4 H), 7.54 (s, broad, 4 H) |
| IIIm | 83 | 90–93 | 1610, 1490, 1300, 1160, 1080, 1015, 850 | 7.15 (dt, $J_v = 4$, $J_g = 40$, 2 H), 7.50 (d, $J = 8$, 4 H), 8.28 (d, $J = 8$, 4 H) |
| IIIn | 83 | 95–98 | 1550, 1380, 1300, 1190, 1110, 1000, 850 | 6.60 (tt, $J_v = 4$, $J_g = 54$, 4 H), 7.44 (t, $J = 2$, 2 H), 8.08 (d, $J = 2$, 4 H) |
| IIIo | 89 | 95–96 | 1590, 1550, 1260, 1170, 1090, 1000, 860, 810 | 6.48 (dt, $J_v = 4$, $J_g = 48$, 4 H), 7.61 (s, broad, 2 H), 8.13 (s, broad, 4 H) |

^aSee footnote a, Table I. ^bRecorded as 10% solutions in the following solvents for the compounds indicated: Unisol, Ia, Ib; trifluoroacetic acid, Ic, Ij, II, Io; DMSO-*d*₆/CDCl₃, Id, Im, In; DMSO-*d*₆, Ie. See remainder of footnote b, Table I.

benzoyl]hydrazine (IIIm); *N,N'*-bis[3,3',5,5'-(1,1,2,2-tetrafluoroethoxy)benzoyl]hydrazine (IIIn); *N,N'*-bis-[3,3',5,5'-(1,1,2-trifluoro-2-chloroethoxy)benzoyl]hydrazine (IIIo).

General Procedure for Preparation of 2,5-Disubstituted-1,3,4-oxadiazoles (I; Scheme III). The *N,N'*-dibenzoylhydrazine (0.00367 mol) and 16 mL of phosphorus oxychloride were added in a 50-mL, round-bottomed flask and the solution refluxed for 20 h. After cooling to room temperature, the solution was poured slowly with stirring into 100 mL of an ice and water mixture. The resulting precipitate was collected, dried

at reduced pressure (Kugelrohr, 1 mm), and recrystallized.

The following compounds are reported in Table III: 2-[4-(1,1,2,2-tetrafluoroethoxy)phenyl]-5-(2,4-dichlorophenyl)-1,3,4-oxadiazole (Ia); 2-[4-(1,1,2-trifluoro-2-chloroethoxy)phenyl]-5-(2,4-dichlorophenyl)-1,3,4-oxadiazole (Ib); 2-[4-(1,1-difluoro-2,2-dichloroethoxy)phenyl]-5-(2,4-dichlorophenyl)-1,3,4-oxadiazole (Ic); 2-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]-5-(2,4-dichlorophenyl)-1,3,4-oxadiazole (Id); 2-[3-(1,1,2-trifluoro-2-chloroethoxy)phenyl]-5-(2,4-dichlorophenyl)-1,3,4-oxadiazole (Ie); 2,5-bis-[3,3'-(1,1,2,2-tetrafluoroethoxy)phenyl]-1,3,4-oxa-

diazole (Ij); 2,5-bis-[3,3'-(1,1-difluoro-2,2-dichloroethoxy)-phenyl]-1,3,4-oxadiazole (Il); 2,5-bis[4,4'-(1,1,2-trifluoro-2-chloroethoxy)phenyl]-1,3,4-oxadiazole (Im); 2,5-bis-[3,3',5,5'-(1,1,2,2-tetrafluoroethoxy)phenyl]-1,3,4-oxadiazole (In); 2,5-bis-[3,3',5,5'-(1,1,2-trifluoro-2-chloroethoxy)phenyl]-1,3,4-oxadiazole (Io).

General Procedure Used in the Insecticide Screen. The wild type strain *Drosophila melanogaster*, Oregon-RC, was used in the screening procedure. One hundred *Drosophila* eggs were placed in a 1 X 9 cm vial containing an 8-mL solution of the egg culture media (a standard yeast, corn meal, agar mixture) and the compound to be screened (dissolved or dispersed in soybean oil). Larvacidal kill was the difference between the number of eggs placed on the media and the number of pupae formed. Total kill was the difference between the number of eggs and the number of living adults.

Registry No. Ia, 114467-42-4; Ib, 114467-43-5; Ic, 114467-44-6; Id, 114467-45-7; Ie, 114467-46-8; Ij, 114467-47-9; Il, 114467-48-0; Im, 114467-49-1; In, 114467-50-4; Io, 114467-51-5; IIa, 114467-26-4; IIb, 114467-27-5; IIc, 114467-28-6; IId, 114467-29-7; IIe, 114467-30-0; IIf, 114467-31-1; IIg, 114467-32-2; IIh, 114467-33-3; III, 114467-34-4; IIj, 114467-37-7; IIk (acid), 70126-48-6; IIk, 114467-38-8; IIk (acid), 403-71-4; III, 114467-39-9; III (acid), 114467-35-5; IIm, 114467-40-2; IIm (acid), 405-43-6; IIn, 114467-41-3; IIn (acid), 70126-49-7; IIo, 114490-28-7; IIo (acid), 114467-36-6; 2,4-Cl₂C₆H₃COCl, 89-75-8; 4-(CHF₂CF₂O)C₆H₄CONHNH₂, 114467-18-4; 4-(CHClCF₂O)C₆H₄CONHNH₂, 114467-19-5; 4-(CHCl₂CF₂O)C₆H₄CONHNH₂, 114467-20-8; 3-(CHF₂CF₂O)C₆H₄CONHNH₂, 114467-21-9; 3-(CHClCF₂O)C₆H₄CONHNH₂, 114504-85-7; 3-(CHCl₂CF₂O)C₆H₄CONHNH₂, 114467-22-0; 3,5-

(CHF₂CF₂O)₂C₆H₃CONHNH₂, 114467-23-1; 3,5-(CHClCF₂O)₂C₆H₃CONHNH₂, 114467-24-2; 3,5-(CHCl₂CF₂O)₂C₆H₃CONHNH₂, 114467-25-3; N₂H₄, 302-01-2.

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A Convenient Synthesis of Amino-Substituted 2-Oxo-1,3,5-triazinium and 1,3,5-Oxadiazinium Salts

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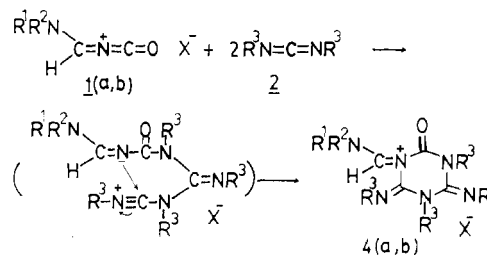
The synthesis of amino-substituted 2-oxo-1,3,5-triazinium hexachloroantimonates **4a,b** and amino-substituted 1,3,5-oxadiazinium hexachloroantimonates **5a-h** and their spectral data are reported.

In the past few years new classes of heterocumulenes have been synthesized and characterized by Jochims and his co-workers (1-7). Especially 1-oxa-3-azabutatrienium salts, **1**, show a wide range of reactivity. As part of our continuing interest in the reactions of these heterocumulenes, I herein report details of the reaction of amino substituted 1-oxa-3-azabutatrienium hexachloroantimonates **1a,b** with diisopropylcarbodiimide (**2**) and dialkylcyanamide **3a-d** to give amino-substituted 2-oxo-1,3,5-triazinium hexachloroantimonates **4a,b** and amino-substituted 1,3,5-oxadiazinium hexachloroantimonates **5a-h**, respectively, in high yields (Schemes I and II). The products are characterized by ¹H NMR, ¹³C NMR, and IR spectroscopy.

Experimental Section

The melting points are uncorrected. Infrared spectra were recorded on a Perkin-Elmer 299 spectrophotometer. ¹H NMR (250 MHz) and ¹³C NMR (63 MHz) spectra were determined at

Scheme I. Reactions of 1-Oxa-3-azabutatrienium Salts **1a,b** with Diisopropylcarbodiimide (**2**)



- 1a:** R¹ = R² = CH₃
1b: R¹ = CH₃; R² = C₆H₅
2: R³ = (CH₃)₂CH
 X = SbCl₆
4a: R¹ = R² = CH₃; R³ = (CH₃)₂CH
4b: R¹ = CH₃; R² = C₆H₅; R³ = (CH₃)₂CH

303 K on a Bruker MW-250 instrument in CD₃CN with TMS as internal standard (Table I). Elemental analyses were performed by CHN-microanalyse, Fakultät Chemie-Universität Konstanz, FRG. The found elemental analyses for carbon, hydrogen, and nitrogen were in good agreement with those calculated and were submitted for review. All experiments were carried out with exclusion of moisture in absolute solvents.