Experimental Section[2]

5,6-Dihydro-7*H*,12*H*-6-carbamyldibenz[*c*,*f*]azocine (1).—A soln of KCNO (3.57 g, 0.044 mole) in H_2O (55 ml) was added to a soln of 5,6-dihydro-7*H*,12*H*-dibenz[*c*,*f*]azocine·HCl[3] (10.8 g, 0.044 mole) in H_2O (6500 ml). After 15 days stirring at room temp, the reaction mixture afforded, when concd, a solid which was recrystd from EtOH to give 1 (6.6 g, 59.4%) as colorless crystals, mp 237–239°. *Anal.* ($C_{16}H_{16}N_2O$) C, H, N.

5,7,12,13-Tetrahydro-6-carbamyldibenz[*c*,*g*]azonine (2).—Compound **2** was obtained similarly in 73.2% yield from 5,7,12,13-tetrahydro-6*H*-dibenz[*c*,*g*]azonine·HCl[4] (12 g, 0.046 mole) and KCNO (3.75 g, 0.046 mole) in H_2O (2000 ml). Colorless crystals from 95% EtOH, mp 194–196°. *Anal.* ($C_{17}H_{18}N_2O$) C, H, N.

1-Cyano-2,3-diphenylaziridine (3).—A soln of BrCN (20.36 g, 0.19 mole) in Et_2O (80 ml) was dropped at 0–5° for 20 min into a soln of *cis*-2,3-diphenylaziridine[5] (31.2 g, 0.16 mole) and Et_3N (19.4 g, 0.19 mole) in Et_2O (400 ml). The mixture was stirred for 4 hr at room temp and then filtered, the cake was repeatedly washed with Et_2O, and the combined filtrates were evapd to dryness. The residue was taken up with hexane and filtered to give **3** (33 g, 94%) as a colorless solid, mp 116–117°. *Anal.* ($C_{15}H_{12}N_2$) C, H, N.

1-Carbamyl-2,3-diphenylaziridine (4).—A mixture of **3** (41.3 g, 0.187 mole), NaOH (75 g), H_2O (130 ml), and dioxane (950 ml) was stirred for 7 days at room temp and then for 24 hr at 50°. The resulting cloudy soln was evapd to dryness under reduced pressure, and the residue was taken up with H_2O and little Et_2O and then recrystd from C_6H_6 to give **4** (11.2 g, 25%) as colorless crystals, mp 158–160°. *Anal.* ($C_{15}H_{14}N_2O$) C, H, N.

(2) Melting points are corrected and were taken on a Büchi capillary melting point apparatus. All compounds were analyzed for C, H, N and anal. results were within ±0.4% of the theoretical values.

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Some Derivatives of 7-Oxabicyclo[2.2.1]heptane-*exo*-*cis*-2,3-dicarboxylic Acid

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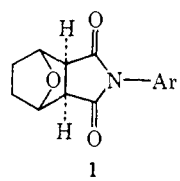
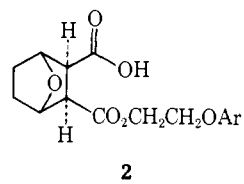
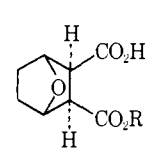
Recently some 7-oxabicyclo[2.2.1]heptane-2,3-dicarboximides (**1**) with anticonvulsant activity were described.[1] Some aryloxyethyl esters **2** were also reported[2] as plant growth regulators. We record herein the preparation of additional examples of **1** and of some mono esters **3**, all of which proved to be highly toxic CNS depressants (Table I).



1



2



3

Experimental Section

***N*-Fluoroarylimides.** 1.—A mixture of equimolar amts of 7-oxabicyclo[2.2.1]heptane-*exo*-*cis*-2,3-dicarboxylic anhydride and the appropriate fluoroaniline was heated without solvent at 150° for 1–2 hr. The cooled residue was then recrystd from EtOH.

Monoesters. 3.—A mixture of anhydride and the appropriate alcohol was heated at 125° for 1–2 hr. The cooled residue was extd with aq Na_2CO_3 and the aq extracts were acidified with HCl. The ppt was collected, washed with H_2O, dried, and recrystd from an appropriate solvent, usually C_6H_6–Skelly B.

The *i*-Pr deriv **3a** was prepared by refluxing the anhydride in *i*-PrOH containing pyridine.

Table I

Compd	R or Ar	Formula[a]	Mp,[b] °C	Approx[c] LD, mg/kg
1a	*o*-FC_6H_4	$C_{14}H_{12}FNO_3$	135–137	1000
1b	*m*-FC_6H_4	$C_{14}H_{12}FNO_3$	136–138	300
1c	*p*-FC_6H_4	$C_{14}H_{12}FNO_3$	168–169	300
3a	$(CH_3)_2CH$	$C_{11}H_{16}O_5$	127–129	300
3b	*o*-$CH_3OC_6H_4CH_2$	$C_{16}H_{18}O_6$	98–100	30
3c	*m*-$CH_3OC_6H_4CH_2$	$C_{16}H_{18}O_6$	127–128	30
3d	*p*-$CH_3OC_6H_4CH_2$	$C_{16}H_{18}O_6$	110–112	10
3e	$C_6H_5CH_2$	$C_{15}H_{16}O_5$	122–124	30
3f	*m*-$ClC_6H_4CH_2$	$C_{15}H_{15}ClO_5$	143–145	30
3g	*p*-$ClC_6H_4CH_2$	$C_{15}H_{15}ClO_5$	158–160	30
3h	*p*-$FC_6H_4CH_2$	$C_{15}H_{15}FO_5$	135–136	10
3i	3,4-$(OCH_2O)C_6H_3CH_2$	$C_{16}H_{16}O_7$	145–147	10

[a] All new compounds described gave elemental analyses for C and H within ±0.4% of the calculated values. Ir and nmr spectra were also in agreement with the assigned structures; in particular, the nmr spectra confirmed the assignment of exo-cis stereochemistry.[1] [b] Uncorr; recorded on a Mel-Temp apparatus. [c] Dose at which fatalities occurred; compds were administered ip to mice.

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Potential Antidiabetics. 7. N^1-(β-Hydroxybenzylmethyl)-3-methyl-4-arylhydrazono-2-pyrazolin-5-ones and N^1-(β-Hydroxybenzylmethyl)-3-methyl-4-arylazo-5-methyl- or -phenylpyrazoles

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A few pyrazoles and related compounds appear to give promising results in antidiabetic tests[1,2] and, therefore, further combinations seem worthwhile studying. This paper describes the synthesis of N^1-(β-hydroxybenzylmethyl)-3-methyl-4-arylhydrazono-2-pyrazolin-5-ones and N^1-(β-hydroxybenzylmethyl)-3-methyl-4-arylazo-5-methyl- or -phenylpyrazoles and also includes the hypoglycemic activity of 3-methyl-4-arylazo-5-phenylisoxazoles.[2]

Biological Results.—On oral administration at various doses (25–100 mg/kg) in fasted guinea pigs for 18 hr prior to and during testing, 4-phenylazo-, 4-(2-nitrophenylazo)-, 4-(3-nitrophenylazo)-, 4-(2-methylphenylazo)-, 4-(2-methoxyphenylazo)-, 4-(3-methoxyphenylazo)-, 4-(4-ethoxyphenylazo)-, 4-(2,5-dichlorophenylazo)-, and 4-(2,6-dichlorophenylazo)-3-methyl-5-phenylisoxazoles essentially displayed no hypoglycemic activity as compared with chloropropamide. After a predetermined time of peak effect the blood was ana-

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