TABLE I

R_f VALUES OF NUCLEOSIDES AND NUCLEOTIDES

Solvent	Paper chromatography				Paper electrophoresis, R_{Up}[e]
	A[a]	B[b]	C[c]	D[d]	
5′-Acetyladenosine	0.58	0.55			
Adenosine	0.51	0.46			0.16
Adenosine-2′,3′ cyclic phosphate	0.46	0.38			0.69
Adenosine 2′-phosphate and 3′-phosphate	0.12	0.20		0.31 (2′) 0.18 (3′)	0.91
5′-Methoxytrityladenosine	0.88	0.79			
5′-Methoxytrityladenosine-2′,3′ cyclic phosphate	0.75				0.71
5′-Methoxytrityladenosine 2′-phosphate and 3′-phosphate	0.45				
5′-Benzoyluridine	0.72	0.73	0.72		
Uridine	0.46	0.49	0.57		0.43
Uridine-2′,3′ cyclic phosphate	0.31	0.43	0.40		0.81
Uridine 2′-phosphate and 3′-phosphate	0.09	0.27	0.11		1.00
Uridyl-(3′→5′)-uridine	0.15		0.26		0.66
Diphenyl phosphate	0.89	0.79			
Benzoic acid	0.79	0.66			

[a] Isopropyl alcohol–concentrated ammonia–water, 7:1:12 (descending). [b] Isopropyl alcohol–1% ammonium sulfate, 2:1 (ascending). [c] Ethanol–1 *N* ammonium acetate, 5:2 (ascending). [d] Saturated ammonium sulfate–1 *M* ammonium acetate–isopropyl alcohol, 79:19:2 (descending). [e] R_f values divided by R_f of uridine 5′-phosphate.

phosphates, which were converted to adenosine 2′- and 3′-phosphate by hydrolysis with 0.1 *N* hydrochloric acid at 30° for 2 hr.

Uridine-2′,3′ Cyclic Phosphate.—The reaction was carried out by essentially the same procedure as described in the case of 5′-O-acetyladenosine, using the following reagents: diphenyl phosphate (75 mg, 0.3 mmole) in 2 ml of dioxane; morpholinophosphorochloridate (122 mg, 0.6 mmole) and 2,6-lutidine (128 mg, 1.2 mmoles) in 1 ml of dioxane; and 5′-O-benzoyluridine (105 mg, 0.3 mmole) in 2 ml of dioxane. The extent of the reaction estimated after paper electrophoresis was 73%. After the reaction, 0.1 *N* ammonium hydroxide (40 ml) was added to the reaction mixture, which was kept for 3 hr at room temperature. Extraction with ether (three 30-ml portions) and evaporation of the water layer gave a solution having TOD_{260} 2640. The solution was applied to a column (34 × 2.2 cm) of DEAE-cellulose (bicarbonate form) and the column was eluted with 0–0.25 *M* triethylammonium bicarbonate (pH 7.5) buffer. Evaporation of the fractions corresponding to uridine-2′,3′ cyclic phosphate gave the nucleotide in 38% yield. This material was compared with an authentic sample by paper chromatography and paper electrophoresis. R_f values are shown in Table I. Ultraviolet absorption was at $\lambda_{\max}^{\mathrm{H_2O}}$ 262 mμ. Hydrolysis with acid or alkaline gave uridine 2′- and 3′-phosphate, which were identical with the authentic samples.

Enzymatic Assay of Uridine-2′,3′ Cyclic Phosphate.—Uridine-2′,3′ cyclic phosphate (triethylammonium salt) obtained as above (15 OD) was dissolved in 0.1 ml of Tris hydrochloride buffer (pH 7, 0.05 *M*), followed by the addition of pancreatic ribonuclease (200 μg) dissolved in 0.2 ml of the buffer. Incubation of this mixture for 24 hr at 37° showed the complete digestion to uridine 3′-phosphate by paper chromatographic tests. A control experiment without addition of RNAase showed no hydrolysis.

Enzymatic Synthesis of Uridylyl-(3′→5′)-uridine.—Uridine-2′,3′ cyclic phosphate (200 OD) was dissolved in 0.3 ml of Tris hydrochloride buffer (pH 7, 0.05 *M*), followed by the addition of uridine (14.6 mg, 60 μmoles) and pancreatic RNAase (3 μg dissolved in 3 μl of buffer). The mixture was incubated for 20 hr at 0–1.° After the reaction, 1.6 ml of chloroform–isoamyl alcohol (1:1 v/v) mixture was added. An aliquot examined by paper chromatography showed a new spot having R_f (A) 0.15 and R_f (C) 0.26 in addition to the spots of uridine, uridine-2′,3′ cyclic phosphate, and uridine 2′- and 3′-phosphate. The newly appeared spot was confirmed as uridylyl-(3′→5′)-uridine by the comparison with an authentic sample.[29]

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[29] Y. Lapidot and H. G. Khorana, *J. Am. Chem. Soc.*, **85**, 1363 (1963).

Aromatic Polyfluoronitroso Compounds

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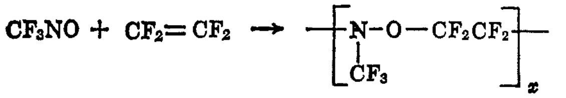
The preparation of 4-nitrosotetrafluorobromobenzene and 4-nitrosotetrafluorobenzoic acid by oxidation of the appropriately substituted anilines with performic acid is reported. The compounds reacted with 1,3-cyclohexadiene to give substituted N-phenyl-2-oxa-3-azabicyclo[2.2.2]oct-5-enes *via* the Diels–Alder reaction.

The reaction of trifluoronitrosomethane and tetrafluoroethylene has been reported to yield a 1:1 copolymer generally referred to as nitroso rubber.[2,3]



$$\mathrm{CF_3NO + CF_2{=}CF_2 \longrightarrow \left[-\underset{\displaystyle CF_3}{\underset{|}{N}}-O-CF_2CF_2- \right]_x}$$

This copolymer can be cured only through the use of polyfunctional amines to yield products of low tensile strength. Polyfluoroaromatic nitroso compounds are therefore of interest as termonomers to provide sites for cross-linking the polymer to higher tensile strength products.

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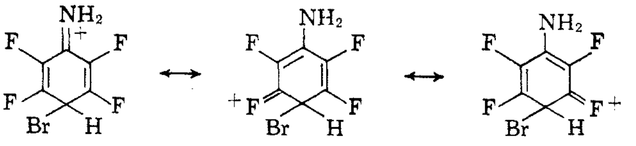
[1] To whom correspondence should be addressed.

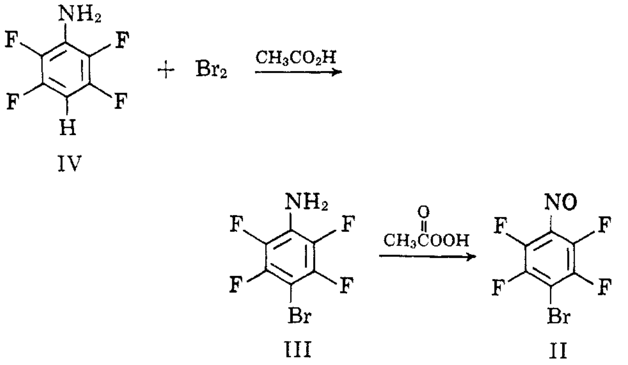
[2] D. A. Barr and R. N. Haszeldine, *J. Chem. Soc.*, 1881 (1955).

[3] G. H. Crawford, D. F. Rice, and B. F. Landrum, *J. Polymer Sci.*, **A1**, 565 (1963).

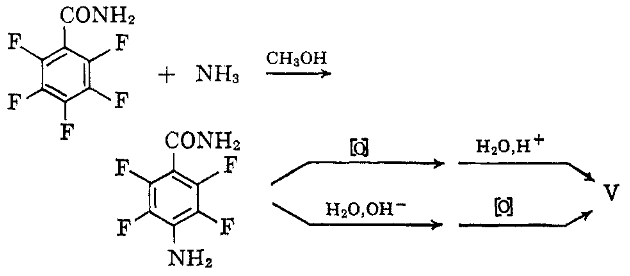
Persulfuric acid or peroxycarboxylic acids have been successfully employed[4] for the oxidation of aromatic amines to nitroso compounds. Performic acid has been reported[5] to be useful for the oxidation of pentafluoroaniline to pentafluoronitrosobenzene (I). We used this procedure to prepare I in 46.6% yield. The product, which is monomeric in the crystalline as well as the liquid or dissolved state, was obtained as blue crystals and was characterized by comparison of its infrared spectrum and melting point with those reported previously.[5]

The potential reactivity of the *p*-bromine atom in 4-nitrosotetrafluorobromobenzene (II) toward nucleophilic reagents indicated that this compound might be another interesting termonomer candidate. The synthesis of II was therefore carried out in 64% yield by the performic acid oxidation of 4-bromotetrafluoroaniline (III). III was obtained in 70% yield by bromination of 2,3,5,6-tetrafluoroaniline (IV) in glacial acetic acid at room temperature. The bromination occurred readily because the strong resonance effect of the fluorine atoms[6] and amino group makes the *para* position in IV highly susceptible to attack by electrophilic reagents. II was obtained as the colorless dimer which is readily converted to the monomer by heat (mp 39–40° to a blue-green liquid) or dissolution in a suitable solvent.





The preparation of 4-nitrosotetrafluorobenzoic acid (V) was explored by the following reaction sequence.



The pentafluorobenzamide (VI) was prepared by a previously reported[7] procedure from pentafluorobenzoyl chloride. Many attempts to prepare 4-aminotetrafluorobenzamide (VII) by the reported[8] reaction of VI with sodium amide in liquid ammonia were unsuccessful. An alternate method[8] which involved treatment of VI with ammonia and methanol, however, resulted in the formation of a 20% yield of VII. When the oxidation of VII with performic acid was conducted, a 31% yield of 4-nitrotetrafluorobenzamide was obtained and no evidence for the formation of a nitroso compound was observed.

Another reaction sequence, which involves hydrolysis of the amide followed by oxidation of the amino acid, was successfully used to prepare V in 28–47% yields. Hydrolysis of VII was carried out with 20% aqueous sodium hydroxide, and 64 to 70% yields of 4-aminotetrafluorobenzoic acid (VIII) were produced. The oxidation reaction of VIII with performic acid was examined and was best carried out by refluxing the reactants in a large volume of methylene chloride for 4 to 6 hr. The products were then separated from each other by utilizing the difference in their distribution coefficients in methylene chloride and water. V was isolated as (1) a pale yellow, crystalline dimer (Va) which was readily converted to monomer on dissolution in methanol, ethanol, ether, or acetone and (2) a colorless, crystalline dimer (Vb) which did not form monomer in solution. Yields of 28 to 34% of Va were obtained by this method while higher yields (38–47%) could be recovered by extraction of the aqueous solution with ether and isolation of the product by silica gel column chromatography. When Va was heated slowly from room temperature, a color change from yellow to brown was observed in the temperature range between 100 and 200°. At 226–227° the material decomposed sharply with the evolution of gas. However, when a capillary tube containing Va was plunged into a 145° bath and the temperature was slowly increased, it was found to melt at 150–151° to a green liquid (which then decomposed). The dimer Vb, on the other hand, darkened above 200° but did not melt below 360°. The infrared spectra of Va and Vb are almost identical. This suggests that dimer Va is the *cis* isomer, which can readily be transformed into monomer, and that Vb is the *trans* isomer which is stabilized by virtue of the coplanarity of the aromatic rings and consequently the delocalization of electrons. A study of nonfluorinated nitroso dimers,[4] in which the structure of these compounds is shown to have the azoxy type linkage, appears to support this supposition.

Bicyclooxazine Derivatives.—Aromatic nitroso compounds are known[9,10] to act as dieneophiles in Diels–Alder reactions with substituted butadienes. A free-radical mechanism has been suggested[10] for the addition reaction. Recently, nitrosobenzene was found[11] to give a Diels–Alder adduct on reaction with 1,3-

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(4) B. G. Gowenlock and W. Luttke, *Quart. Rev.* (London), **12**, 321 (1958).

(5) G. M. Brooke, J. Burdon, and J. C. Tatlow, *Chem. Ind.* (London), 832 (1961).

(6) A. E. Pavlath and A. J. Leffler, "Aromatic Fluorine Compounds," Reinhold Publishing Corp., New York, N. Y., 1962, p 31. Studies of the relative rates of substitution of aryl halides show that fluorine exerts the strongest resonance effect of the halogens. The inductive effect of the highly electronegative fluorine atom is overwhelmed and resonance forms can be written to illustrate the stability of the intermediate in the transition state.

(7) G. M. Brooke, J. Burdon, and J. C. Tatlow, *J. Chem. Soc.*, 802 (1961).

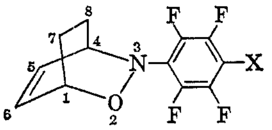
(8) L. J. Belf, British Patent 915,587 (1963).

(9) O. Wichterle, V. Gregor, A Dubansky, and V. Seidl, *Chem. Listy*, **51**, 605 (1957); *Chem. Abstr.*, **51**, 11354*a* (1957).

(10) Yu. A. Arbuzov and T. A. Pisha, *Dokl. Akad. Nauk SSSR*, **116**, 71 (1957); *Chem. Abstr.*, **52**, 6357*d* (1958).

(11) G. Kresze, G. Schulz, and J. Firl, *Angew. Chem.*, **75**, 375 (1963).

cyclohexadiene. We have found that blue-green solutions of I, II, and Va can be "titrated" with 1,3-cyclohexadiene to give colorless solutions of the Diels–Alder adducts IX, X, and XI, respectively. This rapid high-yield reaction is a convenient procedure for the preparation of derivatives of aromatic compounds.



IX, X = F
X, X = Br
XI, X = COOH

The structural assignment was confirmed by H^1 nuclear magnetic resonance. The nmr spectrum showed a multiplet centered at τ 3.38 due to protons at the unsaturated carbon atoms with broad peaks at τ 5.25 and 5.07 which were assigned to the bridgehead protons. An A_2B_2 multiplet with the appearance of an AB quartet gives peaks at τ 8.60, 8.43, 7.78, and 7.63 due to protons on the saturated carbon atoms.

Experimental Section[12]

Pentafluoronitrosobenzene (I).—The method described previously[5] was modified as follows. A mixture of 200 ml of 98% formic acid, 50 ml of 90% hydrogen peroxide, and 600 ml of methylene chloride was stirred while a solution of 50 g (0.27 mole) of pentafluoroaniline (Pierce Chemical Co., mp 34–35°) in 200 ml of methylene chloride was added slowly over a 45-min period at room temperature. The mixture was refluxed for 5 hr. After the addition of 500 ml of water, the dark green mixture was refluxed for 2 hr. The organic solution was separated, washed three times with water, and dried over anhydrous sodium sulfate. Evaporation of the solvent yielded 47.6 g of crude green liquid. The liquid was distilled to yield 28.0 g of blue liquid, bp 50–60° (20 mm). Sublimation of the product afforded 25.1 g (0.128 mole, 46.6%) of blue-green pentafluoronitrosobenzene, mp 42–44° (lit.[5] mp 45°). Recrystallization of this material provided a product with mp 44.5–45°. The infrared spectrum showed a strong absorption at 1550 cm^{-1} indicative of the NO stretching frequency.

4-Bromo-2,3,5,6-tetrafluoroaniline (III).—A solution of 20.0 g (0.121 mole) of 2,3,5,6-tetrafluoroaniline (Pierce Chemical Co., mp 30–32°) in 100 ml of glacial acetic acid was treated with a solution of 8.00 ml (23.4 g, 0.146 mole) of bromine in 20 ml of glacial acetic acid at room temperature in a 500-ml flask. The bromine solution was added at a moderate rate and the resultant mixture was stirred at room temperature for 2 hr. After the mixture was poured into 500 ml of cracked ice, the separated solid was collected by filtration. The yellow solid was washed with water, dried by suction, and recrystallized from pentane to yield 20.8 g (0.0853 mole, 70.5%) of colorless crystals, mp 54–55°. After a second recrystallization from pentane, the melting point was 57–58° (cor).

Anal. Calcd for $C_6H_2BrF_4N$: C, 29.53; H, 0.83; Br, 32.75; N, 5.74. Found: C, 29.57; H, 0.82; Br, 32.80; N, 5.97.

This material was insoluble in concentrated HCl solution, and attempts to prepare the acetamide and benzamide derivatives by the usual method[13] resulted in recovery of unchanged starting materials.

4-Nitrosotetrafluorobromobenzene (II).—A mixture of 120 ml of 98% formic acid and 30 ml of 90% hydrogen peroxide stirred for 15 min at room temperature was added to a solution of 15.0 g (0.062 mole) of 4-bromotetrafluoroaniline and 400 ml of methylene chloride in a 1-l. flask and refluxed for 5 hr. The resulting dark green mixture was then treated with 250 ml of water and refluxed for 30 min. The deep green organic phase was separated, washed with water, and dried over anhydrous sodium sulfate. The solvent was removed *in vacuo*, the residue was taken up in pentane, and the solution was chromatographed on a 2 × 50 cm column of acid-washed alumina (Merck, suitable for chromatographic analysis). After the column was eluted with pentane, the blue-green solution was collected and evaporated to dryness to yield 10.2 g (0.0396 mole, 64.2%) of pale yellow solid, melting at 36–37° to a blue-green liquid. Recrystallization of the material from ethanol produced colorless crystals, mp 39–40° (cor), of 4-nitrosotetrafluorobromobenzene.

Anal. Calcd for C_6BrF_4NO: C, 27.93; Br, 30.98; N, 5.43. Found: C, 28.15; Br, 31.00; N, 5.29.

The infrared spectrum of the blue-green liquid exhibited strong sharp absorptions a 1530, 1510, 1480, and 1360 cm^{-1}. The absorption at 1530 cm^{-1}, which is due to the N=O stretching frequency, is much weaker in intensity in the spectrum of the solid dimer. In addition, the 1360-cm^{-1} absorption is completely absent in the spectrum of the solid.

Pentafluorobenzamide (VI).—The previously described procedure[7] was used to prepare pentafluorobenzamide, mp 148–150° (cor) (lit.[7] mp 150°), in 60% yield by the reaction of ammonia with pentafluorobenzoyl chloride (prepared from pentafluorobenzoic acid and phosphorus pentachloride).

4-Aminotetrafluorobenzamide (VII).—Attempts to prepare 4-aminotetrafluorobenzoic acid and 4-aminotetrafluorobenzamide by the reaction of the acid or amide, respectively, with sodium amide in liquid ammonia by the previously described procedure[8] were unsuccessful. An alternate method of amination was used successfully to prepare VII. A mixture of 50 ml of methanol saturated with ammonia and 5.0 g (0.024 mole) of pentafluorobenzamide contained in a Fischer–Porter pressure reactor was heated for 8 hr in a steam bath. The pressure was observed to be 130 psi. The mixture was cooled and evaporated to dryness, and the product was recrystallized from toluene–methanol to yield 1.05 g (0.0051 mole, 23%) of pink needles, mp 180–182° (lit.[8] mp 171–171.5°), soluble in ethyl acetate. The infrared spectrum was consistent with the assigned structure.

Anal. Calcd for $C_7H_4F_4N_2O$: C, 40.39; H, 1.94; N, 13.46. Found: C, 39.96; H, 1.92; N, 13.23.

4-Aminotetrafluorobenzoic Acid (VIII).—The previously described procedure[8] was modified as follows. A mixture of 50.0 g (0.24 mole) of 4-aminotetrafluorobenzamide and 250 ml of 20% aqueous sodium hydroxide was refluxed for 10 hr. The resulting solution was acidified with concentrated hydrochloric acid and the precipitated acid was collected by filtration. The solid was recrystallized from acetone to yield 32 g (0.15 mole, 64%) of colorless crystals, mp 170–172°. A second recrystallization from CCl_4 gave crystals melting at 176–177° (lit.[8] mp 181–181.5°), pK_a = 3.65. The infrared spectrum confirmed the identification.

Anal. Calcd for $C_7H_3F_4NO_2$: C, 40.21; H, 1.45; N, 6.70; equiv wt, 209. Found: C, 39.66; H, 1.77; N, 6.73; equiv wt, 208.

4-Nitrotetrafluorobenzamide.—A mixture of 7 ml of 98% formic acid, 3 ml of 90% hydrogen peroxide, 1.0 g (0.0048 mole) of 4-aminotetrafluorobenzamide, and 50 ml of ethyl acetate in a 100-ml flask was stirred for 1 hr at room temperature. The solution changed from amber to yellow. Failure to detect a green color indicated that oxidation to the nitro compound had taken place. The mixture was then refluxed (80°) for 2.5 hr and allowed to cool overnight. An additional 1.5 ml of 90% hydrogen peroxide was added and the mixture was refluxed for 2.5 hr. The yellow mixture was poured into 100 ml of 10% sodium carbonate solution and the organic solution was separated, washed with water, and dried over anhydrous sodium sulfate. The solution was evaporated to dryness to yield 0.9 g (79% crude yield) of yellow solid, mp 101–106°. This solid was recrystallized twice from carbon tetrachloride to yield 0.35 g (0.0013 mole, 31%) of colorless plates, mp 110–112°.

Anal. Calcd for $C_7H_2F_4N_2O_3$: C, 35.31; H, 0.85; N, 11.76. Found: C, 35.59; H, 1.03; N, 11.55.

The compound was further characterized by its infrared spectrum, which exhibited absorptions at 1555 and 1360 cm^{-1}.

4-Nitrosotetrafluorobenzoic Acid (V). A.—A mixture of 5.0 g (0.024 mole) of 4-aminotetrafluorobenzoic acid and 250 ml of methylene chloride in a 500-ml flask was treated with a previously prepared solution of 40 ml of 98% formic acid and 10

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[12] All melting points were taken with an Arthur H. Thomas melting point apparatus and are uncorrected unless otherwise noted. The infrared spectra were prepared with the Perkin-Elmer Model 521 spectrophotometer; nmr spectra were recorded with a Varian Associates A-60 spectrometer.

[13] R. L. Shriner and R. C. Fuson, "Identification of Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 1948, p 177.

ml of 90% hydrogen peroxide and refluxed for 5–6 hr. After the addition of 200 ml of water, the mixture was refluxed for an additional 1 hr. The blue-green organic phase was separated, dried over anhydrous sodium sulfate, and evaporated to dryness to yield 1.8 g (0.008 mole, 34%) of pale yellow solid (Va), mp 150–151°, 226–227° dec.

Anal. Calcd for $C_7HF_4NO_3$: C, 37.69; H, 0.45; N, 6.28; equiv wt, 223. Found: C, 37.89; H, 0.73; N, 5.91; equiv wt, 225.

The melting characteristics of Va are worthy of note. Slow heating from room temperature causes a gradual darkening in the color of the solid above 100°. At 226° the material is black and decomposes over a 1° range with the evolution of gas. When a capillary tube containing the product was plunged into an oil bath at 145° and then the temperature was slowly raised, the compound melted at 150–151° to a green liquid which immediately liberated gas. The latter value is probably the correct melting point, whereas the former represents the melting point of another compound apparently formed during the heating process.

The compound exhibits absorptions at 780 mμ (ϵ 34.3), 302 mμ (sh), and 281 mμ (ϵ 1.02×10^4) in methanol.

The infrared spectrum of Va exhibited a CO absorption at 1715 cm^{-1} and an NO absorption at 1555 cm^{-1}.

B.—A solution of performic acid was prepared by stirring 20 ml of 98% formic acid with 5 ml of 90% hydrogen peroxide for 15 min at room temperature. This solution was added to a stirred suspension of 2.0 g (0.010 mole) of 4-aminotetrafluorobenzoic acid in 100 ml of methylene chloride and the mixture was refluxed for 4 hr. After the addition of 50 ml of water, the green mixture was refluxed for 1 hr. The organic phase was separated and the aqueous solution was extracted with ether. All of the green color was transferred from the aqueous phase to the organic layer. The organic solutions were combined and dried over anhydrous sodium sulfate. After removal of the solvent *in vacuo*, the residue was taken up in ether and the solution was chromatographed on a carefully packed column of silica gel (Baker) which was kept at 15° by a water-cooled jacket. The deep-blue green solution was evaporated to dryness to yield 1.0 g (0.0044 mole, 47%) of pale yellow dimer (Va), mp 149–150°, 223–225° dec.

In one experiment the former method (A) was used and the aqueous solution produced during work-up of the reaction mixture was extracted with ether. After the ether extract was dried over anhydrous sodium sulfate, the solvent was removed *in vacuo* and the resulting yellow paste was treated with water. The concentrated aqueous solution was then extracted with methylene chloride containing a small amount of ether, and the organic solution was dried over anhydrous sodium sulfate. Removal of the solvent *in vacuo* gave a 30% yield of yellow solid (Vb), mp >360°, which had an infrared spectrum almost identical with that of Va.

Anal. Calcd for $C_7HF_4NO_3$: C, 37.69; H, 0.45; N, 6.28; mol wt, 446. Found: C, 38.29; H, 0.57; N, 6.44; mol wt, 448.

3-(4′-Bromotetrafluorophenyl)-2-oxa-3-azabicyclo[2.2.2]oct-5-ene (X).—A solution of 1.0 g (0.0039 mole) of 4-bromotetrafluoronitrosobenzene in 50 ml of methylene chloride was treated with 3.0 ml (2.5 g, 0.032 mole) of 1,3-cyclohexadiene (Aldrich Chemical Co.). The solution turned from deep blue-green to colorless immediately. The solvent was evaporated under vacuum to yield 1.20 g (0.0036 mole, 92%) of the adduct, mp 101–103°. The infrared spectrum and the H^1 nmr spectrum confirm the structural assignment.

Anal. Calcd for $C_{12}H_8BrF_4NO$: C, 42.63; H, 2.39; Br, 23.64; N, 4.14. Found: C, 42.65; H, 2.30; Br, 24.07; N, 4.32.

3-Pentafluorophenyl-2-oxa-3-azabicyclo[2.2.2]oct-5-ene (IX).—Pentafluoronitrosobenzene was allowed to react with 1,3-cyclohexadiene as above to give a 90.5% yield of the Diels–Alder adduct, mp 67.5–69°.

Anal. Calcd for $C_{12}H_8F_5NO$: C, 52.00; H, 2.91; N, 5.05. Found: C, 52.30; H, 2.99; N, 4.93.

The H^1 nmr spectrum of this compound is identical with that of the analogous bromine-containing compound and the infrared spectrum is also quite similar.

3-(4′-Carboxytetrafluorophenyl)-2-oxa-3-azabicyclo[2.2.2]oct-5-ene (XI).—4-Nitrosotetrafluorobenzoic acid was allowed to react with 1,3-cyclohexadiene as above to give an 86% yield of the Diels–Alder adduct, mp 174–176° dec.

Anal. Calcd for $C_{13}H_9F_4NO_3$: C, 51.49; H, 2.99; N, 4.62. Found: C, 50.89; H, 3.07; N, 4.53.

The infrared spectrum was very similar to that of the analogous bromine- and fluorine-containing compounds. The nmr spectrum was not obtained because of the lack of a suitable solvent.

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