give 0.5 g (95%) of colorless, viscous oil, bp 120–130° (bath) (0.1 mm). Gas chromatographic examination utilizing a 20 ft × 1/8 in. column packed with 10% FFAP on Aeropak 30, 80–100, indicated less than 5% of the cis isomer **8**. The major product impurity (~10–15%) has been tentatively identified as bornyl isomer **10** (trans). The trans isomer **7** on further purification (FFAP, 200°) exhibited the following properties: $n^{20}$D 1.5115 (lit.[3] $n^{20}$D 1.5112); ir (film) 2.98, 7.19, 10.3 μ; nmr ($CDCl_3$) τ 6.05 (m, 1, CHOH), 7.33 (s, 1, OH), 9.10–9.40 (complex, $CH_3$'s).

*Anal.* Calcd for $C_{16}H_{28}O$: C, 81.29; H, 11.94. Found: C, 81.4; H, 12.0.

The 3,5-dinitrobenzoate derivative after several crystallizations exhibited mp 101–103° (lit.[3] 107–108°). Iridium tetrachloride[11b] could be used in place of chloroiridic acid with comparable results.

**Registry No.**—**4**, 26988-38-5; 3,3′-dibenzyloxybiphenyl, 26988-39-6; **6**, 26988-40-9; **6** semicarbazone, 24739-52-4; **7**, 24739-41-1.

**Acknowledgments.**—The technical assistance of Mr. Kerry M. Fitzpatrick on part of this work is gratefully acknowledged.

## 5,6-Dibromoacenaphth[5,6-*cd*]-1,2-oxathiole 2,2-Dioxide. A Potential Sulfene Precursor

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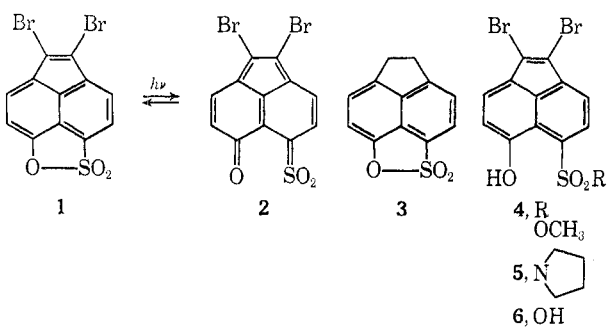
The evidence for a sulfene intermediate generated through photochemical processes is limited.[1-4] We report the synthesis of 5,6-dibromoacenaphth[5,6-*cd*]-1,2-oxathiole 2,2-dioxide (**1**) and adduce evidence that implicates the ketosulfene intermediate (**2**) in photochemical reactions.




Acenaphthene sultone (**3**),[5,6] was treated with 4 equiv of NBS, and the product was isolated in the usual manner.[7] After recrystallization from acetonitrile, a 62% yield of bright red needles of **1**, mp >300°, was obtained. An ethereal solution of **1** when treated with excess pyrrolidine instantly produced an intense purple solution. Evaporation of the ether gave a purple solid that upon heating to 85° (1 mm) in an Abderhalden changed to a deep red solid. Preparative tlc of the substance yielded compound **5**, mp 180–181°. The rapid reaction of **1** with pyrrolidine compared to the slow reaction of **3** with pyrrolidine[8] suggests the existence of ring strain in the ground state of **1**. The purple color is undoubtedly due to the formation of the anion of **5** because the color can be reversibly generated by repeated acid and base treatment of **5**.[9]

A 200-mg sample of **1** was irradiated in 500 ml of absolute methanol using the standard Hanovia 450-W immersion apparatus, Pyrex filter, and continuous nitrogen purge, and maintaining a temperature of 18–20°. A portion of the original reaction mixture was set aside in an opaque container. Periodic analysis of the irradiation reaction by tlc showed that compound **1** disappeared within 30 min. The control mixture maintained in the dark showed no change during the same interval. Removal of the methanol from the irradiated mixture by vacuum rotary evaporation produced 170 mg of deep red **4** that after crystallizing from hexane had mp 107–108°.

A methanolic solution of **1** was allowed to stand undisturbed for 2 weeks in an opaque container. Vacuum rotary evaporation of this solution left a brown residue whose aqueous solution was acidic to Hydrion paper[10] and formed a precipitate upon treatment with $BaCl_2$ solution. Although the brown residue was not further characterized, its properties reflect those to be associated with **6**.

Reaction of **1** with methanolic sodium methoxide followed by neutralization with acid produced a deep red compound whose physical and chemical properties were identical with those of compound **4**. The SN2 ring-opening process of sultones is well documented.[11,12] Thus, this ground-state reaction verifies the structure of **4**.

The differences between ground-state and excited-state solvolysis reactions of **1** are obvious. Although a variety of reactive species in the excited state can be envisioned, we currently favor the ketosulfene **2** as a reactive intermediate derived from an excited singlet-state process. Intermediate **2** is structurally analogous to the proposed diketene intermediates derived from the excited-state chemistry of pyracyloquinone.[13,14]

Additional corroborative evidence for our proposal is a comparison of the irradiation reaction of **1** with acrylonitrile to the irradiation of 1,2-dibromoacenaphthylene in acrylonitrile. No acrylonitrile polymer was found when 20 mg of **1** was irradiated in 5 ml of acrylonitrile through Pyrex during an 8-hr interval. Under similar conditions 1,2-dibromoacenaphthylene causes

(1) J. F. King, P. de Mayo, E. Morkved, A. B. M. A. Sattar, and A. Stoessel, *Can. J. Chem.*, **41**, 100 (1963).

(2) J. F. King and T. Durst, *ibid.*, **44**, 1859 (1966).

(3) R. J. Mulder, A. M. van Leusen, and J. Strating, *Tetrahedron Lett.*, 3057 (1967).

(4) J. L. Charlton and P. de Mayo, *Can. J. Chem.*, **46**, 55 (1968).

(5) The rigorous *Chemical Abstracts* name for **3** is 5,6-dihydroacenaphth-[5,6-*cd*]-1,2-oxathiole 2,2-dioxide.

(6) M. T. Bogert and R. B. Concklin, *Collect. Czech. Chem. Commun.*, **5**, 187 (1933).

(7) This procedure is similar to that for the synthesis of 1,2-dibromoacenaphthylene: B. M. Trost and D. R. Britelli, *J. Org. Chem.*, **32**, 2620 (1967).

(8) Compound **3** was quantitatively recovered after standing for 1 hr admixed with an ethereal solution of excess pyrrolidine.

(9) This result is in accord with the observations[1] made by de Mayo and coworkers upon structurally similar compounds.

(10) Under similar conditions de Mayo and coworkers[1] found "acidic" products resulting from reaction of their sultones.

(11) A. Mustafa, *Chem. Rev.*, **54**, 195 (1954).

(12) O. R. Zaborsky and E. T. Kaiser, *J. Amer. Chem. Soc.*, **92**, 860 (1970).

(13) B. Trost, *ibid.*, **91**, 918 (1969).

(14) F. M. Beringer, R. E. K. Winter, and J. A. Castellano, *Tetrahedron Lett.*, 6183 (1968).

extensive polymerization of acrylonitrile.[15] If 1 forms an excited triplet state, its efficiency for initiating polymerization of acrylonitrile is extremely low.

## Experimental Section

**General.**—Melting points were taken on a Fisher-Johns hot stage and are uncorrected. Ultraviolet and visible spectra were recorded on Beckman DU-2 and DBG spectrophotometers. Infrared spectra were determined on a Perkin-Elmer Model 337 spectrophotometer. Nuclear magnetic resonance spectra were recorded on a Varian Model T-60 spectrometer in hertz (Hz) with reference to internal tetramethylsilane. Elemental analyses were determined by Galbraith Laboratories.

**Sodium Acenaphthene-5-sulfonate.**—To a mixture of 460 g (3.0 mol) of acenaphthene and 2 l. of methylene chloride in a three-necked flask, equipped with overhead stirrer, thermometer, and addition funnel was added dropwise with stirring and ice cooling 349.5 g (3.0 mol, 198 ml) of chlorosulfonic acid. The addition rate was adjusted to maintain the reaction temperature at 0–5°. When the addition was complete, 500 ml of petroleum ether was added, the mixture was filtered, and the white precipitate was washed with 250 ml of petroleum ether. The white solid was dissolved in water, neutralized with solid sodium carbonate, and treated with 600 ml of saturated sodium chloride solution. The mixture was cooled in a refrigerator overnight and the resulting white solid was collected by vacuum filtration. The solid was washed with 250 ml of saturated sodium chloride solution and dried at 110° for 24 hr: yield 465 g (1.82 mol) (62%); mp >300°;[16] ir (KBr) 3000 (C–H) (w), 2900 (m), $SO_2$ 1185 (s), aromatic C–H 1221 (m), 1110 (m), 1100 (m), 1052 (m), 1028 (m), 835 $cm^{-1}$ (m).

**Sodium 6-Nitroacenaphthene-5-sulfonate.**—To a suspension of 465 g (1.82 mol) of pulverized sodium acenaphthene-5-sulfonate and 1 l. of glacial acetic acid, contained in a three-necked flask, fitted with overhead stirrer, thermometer, and addition funnel, was added dropwise, 240 ml of red fuming nitric acid, while maintaining the reaction temperature at 15–16°. After the addition was complete, some material remained suspended, and the mixture was stirred at 0–10° for 15 min. The mixture was poured onto 2 l. of crushed ice and filtered, and the filtrate treated with approximately 30 l. of saturated sodium chloride solution until precipitation was complete. The resulting yellow solid was filtered, dissolved in 500 ml of hot water, precipitated hot with excess saturated sodium chloride solution, and collected by vacuum filtration: yield 285 g (0.95 mol) (54%); mp >300°;[16] ir (KBr) 2920 (C–H) (w), $SO_2$ 1200 (s), $NO_2$ 1510 (s), 1310 (s), 1112 (aromatic C–H) (w), 1040 (w), 1025 (w), 825 $cm^{-1}$ (w).

**6-Aminoacenaphthene-5-sulfonic Acid.**—A solution of 15 g (54 mmol) of sodium 6-nitroacenaphthene-5-sulfonate in 150 ml of water, contained in a 250-ml erlenmeyer flask, was treated with approximately 0.1 g of palladium on charcoal, and 2.5 g (66 mmol) of powdered sodium borohydride was added portionwise, at room temperature, over a period of 1 hr. The mixture which was originally yellow turned dark brown and was filtered to remove the palladium/charcoal; the filtrate was acidified with concentrated hydrochloric acid. The light tan solid was recovered by suction filtration; yield 4.5 g (17 mmol) (31%); mp >300°;[16] ir (KBr) 2930 (C–H) (m), $H_3N$ 2630 (w), 1175 ($SO_2$) (s), 1115 (aromatic C–H) (w), 1055 (m), 1025 (m), 827 $cm^{-1}$ (m).

**Acenaphthene-5,6-sultone.**—To a mixture of 10 ml of concentrated hydrochloric acid, 10 ml of ice, and 2.5 g (10 mmol) of 6-aminoacenaphthene-5-sulfonic acid, in a 125-ml erlenmeyer flask, was added dropwise, with stirring and ice cooling, a solution of 0.69 g (10 mmol) of sodium nitrite in 10 ml of water. When the addition was complete, the mixture was allowed to stand for 10 min and was then filtered. The filtrate was treated with enough sulfamic acid to destroy the excess nitrous acid. The solution was heated to boiling until evolution of nitrogen ceased, and the crude sultone precipitated from the hot solution. After cooling, the white solid was recovered by vacuum filtration, washed with 25 ml of water, and recrystallized from 5 ml of 95% ethanol: yield 0.07 g (0.3 mmol) (3%); mp 176.0–176.5° (lit.[6] mp 173°); ir (KBr) 3070 (C–H) (w), 2920 (w), $SO_2$ 1185 (s), 1208 (aromatic C–H) (m), 1092 (m), 1070 (w), 1035 (w), 834 $cm^{-1}$ (m); nmr $\delta$ ($CDCl_3$) 3.57 (s, 4, $ArCH_2$), 7.02 (d, 1, $J$ = 7.0 Hz), 7.35 (d, 1, $J$ = 7.0), 7.55 (d, 1, $J$ = 7.8), 7.92 ppm (d, 1, $J$ = 7.8); uv ($CH_3OH$) 320 nm ($\epsilon$ 4500), 242 (27,000), 215 (19,500).

**1,2-Dibromoacenaphthylene-5,6-sultone.**—A solution of 0.3 g (1.29 mmol) of acenaphthene-5,6-sultone in 25 ml of carbon tetrachloride was heated to reflux for 15 min, and 0.92 g (5.2 mmol) of freshly recrystallized $N$-bromosuccinimide and approximately 50 mg of dibenzoyl peroxide were added to the hot solution. The mixture was refluxed for 4.5 hr, cooled in an ice bath, and filtered. The solvent was removed from the filtrate by rotary evaporation. The residue was crystallized from 5 ml of chloroform, and recrystallized from 8 ml of acetonitrile; yield 0.31 g (0.81 mmol) (62%); mp >300°; ir (KBr) 3050 (C–H) (w), C=C 1630 (m), $SO_2$ 1190 (s), 1222 (aromatic C–H) (m), 1125 (m), 1040 (w), 1010 (w), 832 $cm^{-1}$ (m); nmr $\delta$ ($CDCl_3$) 6.94 (d, 1, $J$ = 7.8 Hz), 7.64 (d, 1, $J$ = 7.8) 7.78 (d, 1, $J$ = 7.0), 7.98 ppm (d, 1, $J$ = 7.0); uv ($C_6H_{12}$) 450 nm ($\epsilon$ 1140), 370 (8520), 363 (8470), 354 (11,600), 347 (10,500), 335 (13,200), 235 (33,000). *Anal.* Calcd for $C_{12}H_4Br_2SO_3$: C, 37.14; H, 1.04; Br, 41.19; S, 8.26. Found: C, 37.27; H, 1.01; Br, 41.31; S, 8.15.

**Control Reaction. 1,2-Dibromoacenaphthylene-5,6-sultone in Moist Ether.**—A solution of 5.48 mg (14 $\mu$mol) of 1,2-dibromoacenaphthylene-5,6-sultone in 75 ml of moist ether was allowed to stand in the dark, and the progress of the reaction was followed through visible–ultraviolet spectroscopy. During a 2-day period, a peak at 338 nm slowly diminished in intensity while a peak at 332 nm increased. At the same time, two sets of doublets, one at 384 and 370 nm, and the second at 364 and 354 nm, joined to form a lower intensity shoulder at 373 nm and a broad peak at 360 nm. The very broad peak at 450 nm did not shift but its intensity increased in proportion to the other peaks.

**Control Reaction. 1,2-Dibromoacenaphthylene in Moist Ether.**—A solution of 4.8 mg (15 $\mu$mol) of 1,2-dibromoacenaphthylene in 75 ml of moist ether was allowed to stand in the dark. The spectrum of the solution was periodically monitored and during 2 weeks remained unchanged.

**Control Reaction. 1,2-Dibromoacenaphthylene-5,6-sultone in Methanol.**—A solution of 10.46 mg (27 $\mu$mol) of 1,2-dibromoacenaphthylene-5,6-sultone in 75 ml of methanol was allowed to stand in the dark, and the progress of the reaction was followed by visible–ultraviolet spectroscopy. During a 2-week interval the peak at 334 nm slowly disappeared, while a peak at slightly lower intensity appeared at 332 nm. A peak at 256 nm, during the same period, slowly shifted to 360 nm and gained in relative intensity. A slightly lower intensity peak at 370 nm initially grew in intensity while shifting to 375 nm. As the reaction proceeded, however, the relative intensity at 375 nm decreased. The broad, low intensity peak at 450 nm remained at the same position, but its relative intensity increased. The solvent was removed from the solution by rotary evaporation. The dark brown solid dissolved readily in 3 ml of water to form a solution whose resulting pH was approximately 2. Treatment of the aqueous solution with barium chloride immediately gave a precipitate.

**Photolysis of 1,2-Dibromoacenaphthylene-5,6-sultone in Methanol.**—A solution of 0.2 g (0.52 mmol) of 1,2-dibromoacenaphthylene-5,6-sultone in 500 ml of methanol was purged with dry nitrogen for 15 min and irradiated for 40 min in a quartz immersion apparatus, equipped with a magnetic stirrer and a Hanovia 450-W medium-pressure mercury lamp and fitted with a Pyrex filter. The progress of the reaction was followed by tlc. The solvent was removed from the reaction mixture by rotary evaporation and the residue treated with 20 ml of chloroform. The chloroform solution was filtered, the filtrate rotary evaporated, and the bright red solid crystallized from hexane: yield 0.17 g (0.40 mmol) (77%); mp 107.0–107.5°; ir ($CCl_4$) OH 3290 (m), CH 2950) (w); ir (KBr) 1620 (C=C) (m), $SO_2$ 1165 (s), ArH 1180 (m), 1105 (m), 1038 (m), 995 (w), 820 $cm^{-1}$ (m); nmr $\delta$ ($CDCl_3$) 3.82 (s, 3, $OCH_3$), 7.09 (d, 1, $J$ = 8.0 Hz), 7.28 (s, 1, OH), 7.62 (d, 1, $J$ = 8.0), 7.70 (d, 1, $J$ = 8.0), 8.32 ppm (d, 1, $J$ = 8.0); uv ($C_6H_{12}$) 480 nm ($\epsilon$ 1290), 373 (6750), 367 (6560), 355 (8210), 335 (9750), 289 (5660), 247 (22,900), 207 (13,800). *Anal.* Calcd for $C_{13}H_8Br_2O_4S$: C, 37.17; H, 1.92; Br, 38.04; S, 7.63. Found: C, 37.31; H, 1.91; Br, 38.29; S, 7.83.

**Reaction of 1,2-Dibromoacenaphthylene-5,6-sultone and Sodium Methoxide.**—A solution of 1.2 mg (52.8 $\mu$g-atoms) of sodium dissolved in 2 ml of dry methanol was added to a solution of 20.5 mg (52.8 $\mu$mol) of 1,2-dibromoacenaphthylene-5,6-

(15) Private communication from Mr. Richard Hall of these laboratories. See also the report by B. F. Plummer, and R. A. Hall, *Chem. Commun.*, 44 (1970).

(16) No physical constants were reported for this compound.[6]

sultone in 40 ml of dry methanol. The mixture, which turned dark blue, was stirred at room temperature for 15 min. Upon acidification with hydrogen chloride the solution turned red. After removal of the solvent by rotary evaporation, the residue was chromatographed with ethyl acetate on Chrom AR-1000. A fraction moving with the solvent front was recovered as a dark red solid that had melting point, ir, and nmr identical with those of the red photoproduct (4) described above. A small second fraction which was difficult to elute was not characterized.

**Reaction of 1,2-Dibromoacenaphthylene-5,6-sultone and Pyrrolidine.**—A solution of 75.8 mg (0.195 mmol) of 1,2-dibromoacenaphthylene-5,6-sultone in 100 ml of dry ether was treated with 70 mg (0.975 mmol) of pyrrolidine at room temperature and the solution immediately turned a dark purple. The ether was removed from the solution by rotary evaporation. The residue was heated at 85° *in vacuo* for 4 hr and the solid chromatographed on Chrom AR-1000 using benzene to elute the bright red product: yield 72.4 mg (0.158 mmol) (81%); mp 180–181°; ir (KBr) 3190 (OH) (m), 2920 (CH) (w), 1620 (C=C) (m), 1140 ($SO_2$–N) (s), 1175 (ArH) (w), 1095 (w), 1060 (w), 1025 (w), 835 $cm^{-1}$ (w); nmr $\delta$ ($CDCl_3$) 1.77 (m, 4, $CH_2$), 3.25 (m, 4, $NCH_2$), 6.98 (d, 1, $J$ = 7.4 Hz), 7.52 (d, 1, $J$ = 7.4), 7.65 (d, 1, $J$ = 7.4), 8.21 (d, 1, $J$ = 7.4), 10.73 ppm (s, 1, O–**H**); uv ($C_6H_{12}$) 470 nm ($\epsilon$ 1320), 377 (7390), 359 (9040), 335 (9280), 289 (5910), 248 (22,400), 208 (21,400). *Anal.* Calcd for $C_{16}H_{13}NBr_2O_3S$: C, 41.85; H, 2.85; N, 3.05; Br, 34.81; S, 6.98. Found: C, 42.00; H, 2.99; N, 2.93; Br, 34.80; S, 6.73.

**Photolysis of 1,2-Dibromoacenaphthylene-5,6-sultone in Acrylonitrile.**—A solution of 20.0 mg (51.5 $\mu$mol) of 1,2-dibromoacenaphthylene-5,6-sultone in 5 ml of freshly distilled acrylonitrile was irradiated in a Pyrex container for 8 hr, employing a Hanovia 450-W medium-pressure mercury lamp. A small amount of solid precipitated during the irradiation. Upon removal of the remaining acrylonitrile, a red crystalline material was recovered which had infrared absorptions identical with 1,2-dibromoacenaphthylene-5,6-sultone. A small amount of a dark oil was also obtained whose structure was not identified.

**Control Reaction. Acenaphthene-5,6-sultone and Pyrrolidine in Ether.**—To a solution of 20.0 mg (0.086 mmol) of acenaphthene-5,6-sultone in 100 ml of dry ether was added 0.5 ml (0.43 g, 5.98 mmol) of pyrrolidine. Monitoring of the solution by tlc indicated that no reaction occurred during a period of 1 hr.

**Registry No.**—**1**, 26988-41-0; sodium acenaphthene-5-sulfonate, 26988-42-1; sodium 6-nitroacenaphthene-5-sulfonate, 26988-43-2; 6-aminoacenaphthene-5-sulfonic acid, 26988-44-3; 1,2-dibromoacenaphthylene-5,6-sultone with methanol, 26988-49-8; 1,2-dibromoacenaphthylene-5,6-sultone with pyrrolidine, 26988-50-1.

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## Reaction of Nitroprusside with Amines[1]

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In the course of our studies of pentacyanoferrates ($Fe(CN)_5X^{3-}$), we came across a report which described the preparation of aminepentacyanoferrates ($Fe(CN)_5NH_2R^{3-}$) by reaction of nitroprusside ($Fe(CN)_5NO^{2-}$) with primary amines.[2] The reaction of nitroprusside with ammonia and amines was, in fact, first studied by Hofmann and Manchot who also noticed that a gas (presumably nitrogen) was evolved.[3] However, although 2 mol of amine is consumed for each mole of complex produced, no reports have appeared on the *organic* products of this reaction.

$$2RNH_2 + Fe(CH)_5NO^{2-} \longrightarrow Fe(CN)_5NH_2R^{3-} + N_2 + \text{organic products}$$

We have found that the organic products (Table I)

TABLE I
PRODUCTS OF REACTION OF NITROPRUSSIDE WITH AMINES

| Amine | Products (yield, %)[a] | Additional products (yield, %) (in air)[b,c] |
|---|---|---|
| Benzylamine | Benzyl alcohol (120) | Benzonitrile (20), benzaldehyde (trace) |
| Allylamine | Allyl alcohol (38) | Acrylonitrile (trace) |
| Cyclohexylamine | Cyclohexanol (120), cyclohexene (9) | Cyclohexanone (3) |
| 2-Octylamine | 2-Octanol (70), 1- and 2-octene (16) | 2-Octanone (10) |
| 1-Butylamine | 1-Butanol (42), 2-butanol (8) | ...[d] |
| Diethylamine | *N*,*N*-Diethyl-*N*-nitrosamine (44) | |

[a] Yields are based on a stoichiometry of 2 amine: 1 nitroprusside; formation of some $Fe(CN)_5OH_2^{3-}$ in place of $Fe(CN)_5NH_2R^{3-}$, however, may also occur; *cf.* yields for benzylamine and cyclohexylamine. [b] Yields of oxidized products varied with pH and with concentration of nitroprusside; average yields are reported. Yields of nitrosation products (in air) were *ca.* 10% lower than under nitrogen. [c] Infrared spectra of all product mixtures showed weak absorptions at *ca.* 1640 $cm^{-1}$ (>C=N–). [d] The expected oxidation product, *n*-butyraldehyde, reacts with nitroprusside; see ref 10.

are substances derived from *N*-nitrosamines, which indicates that nitroprusside functions as a nitrosating agent. Moreover, nitroprusside is unique in being a nitrosating (and deaminating) agent which is stable in *alkaline aqueous solution. Thus, the deamination of benzylamine can be carried out at an initial pH as high as 12.7.* At higher pH's, nitroprusside is destroyed, according to[4]

$$Fe(CN)_5NO^{2-} + 2OH^- \longrightarrow Fe(CN)_5NO_2^{4-} + H_2O$$

Primary amines give deaminated products (alcohols and olefins), while a secondary amine gives the *N*-nitrosamine. Tertiary and aromatic amines are largely inert. The deaminations are of interest since they probably involve generation of diazonium and carbonium ions in *alkaline solution.*

Moss[5] recently studied the reactions of diazonium ions (prepared from hydrolysis of diazotates) in alkaline solution. He found that the diazotates prepared from primary carbinamines gave predominantly diazoalkanes on hydrolysis, *e.g.*

$$C_4H_9N{=}N{-}O^- \xrightarrow{H_2O} C_4H_9N{\equiv}N^+OH^- \longrightarrow C_3H_7CHN_2 + H_2O$$

We have not observed diazoalkanes with nitroprusside, probably because the hydroxide ion concentration is

(1) Taken from the M.S. Theses of M. A. G. and M. C. N., Boston College, 1970.

(2) D. J. Kenney, J. P. Flynn, and J. B. Gallini, *J. Inorg. Nucl. Chem.*, **20**, 75 (1961).

(3) (a) K. A. Hofmann, *Justus Liebigs Ann. Chem.*, **312**, 1 (1900); (b) W. Manchot and P. Woringer, *Ber.*, **46**, 3514 (1913).

(4) J. H. Swinehart and P. A. Rock, *Inorg. Chem.*, **5**, 573 (1966).

(5) R. A. Moss, *J. Org. Chem.*, **31**, 1082 (1966).