100-52-7; 9, 55177-35-0; 10, 4120-68-7; anthracene, 120-12-7; 7*H*-benz[*de*]anthracen-7-one, 82-05-3; benzil, 134-81-6; 1′-acetonaphthone, 941-98-0; biphenyl, 92-52-4; benzophenone, 119-61-9.

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Hydrolytic and Photolytic Degradation of Flurochloridone

Lydia L. Chang,* Kuo-Shin Lee, and C. K. Tseng

Hydrolytic and photolytic studies of flurochloridone, 3-chloro-4-(chloromethyl)-1-[3-(trifluoromethyl)phenyl]-2-pyrrolidinone, have been completed in buffered aqueous solutions. Flurochloridone hydrolysis rates were measured as a function of pH at 25 and 40 °C. No hydrolysis occurred at pH 5, 7, or 9 at 25 °C or at pH 5 at 40 °C. Appreciable hydrolysis occurred under the remaining conditions; at 40 °C at pH 7 and 9 the observed half-lives were 190 and 140 days, respectively. Five hydrolytic degradation products were identified. Analysis of radioactivity indicated that approximately 96% of the radiocarbon was recovered. Photolysis rates were measured at pH 7 at 25 °C, conditions under which negligible hydrolysis occurred. The pseudo-first-order photolytic half-life was 4.3 days. Six photolytic degradation products were identified. Analysis of radioactivity accounted for 83% of the initial radiocarbon.

Flurochloridone, 3-chloro-4-(chloromethyl)-1-[3-(trifluoromethyl)phenyl]-2-pyrrolidinone (1), is a selective herbicide reported to reduce chlorophyll and carotenoid production in wheat (*Triticum aestivum* var. L. Mericopa) and corn (*Zea mays* var. L. Merit) (Devlin et al., 1979). The herbicide mode of action of flurochloridone was reported by Lay and Niland (1983) and Lay et al. (1985).

The present investigation was undertaken to study the hydrolysis and photolysis processes of flurochloridone and characterize the corresponding degradation products. The information developed in this study will aid in understanding the behavior of the compound in the environment.

EXPERIMENTAL SECTION

Chemicals. Flurochloridone and [3-^{14}C]flurochloridone were produced by Stauffer Chemical Co., Richmond, CA. Technical flurochloridone typically consists of 3 parts trans isomer and 1 part cis isomer. The structural assignment of the isomeric components was reported earlier (Tseng and Gless, 1983). Radiolabeled flurochloridone, with a specific activity 8.5 mCi/mM, was purified by TLC using ethyl ether–pentane (1:3, v/v) as the developing solvent to give a radiopurity of 98%.

Stauffer Chemical Company, Western Research Center, Richmond, California 94804-0023. (Now part of the ICI group of companies.)

1,5-Dihydro-5-methoxy-4-methyl-1-[3-(trifluoromethyl)phenyl]pyrrol-2*H*-one. To a solution of 9.3 g (0.03 mol) of flurochloridone (1) in 150 mL of methanol was added dropwise 14.0 g of 25% sodium methoxide (0.065 mol) in methanol with stirring. The mixture was heated at 60 °C for 3 h, cooled, and filtered. The filtrate was concentrated to a brown oil on a rotary evaporator. The oil was redissolved in 100 mL of methylene chloride, washed with 50 mL of 0.5 M HCl followed by 50 mL of water, dried over anhydrous sodium sulfate, and concentrated. The resulting residue was purified by column chromatography using hexane–chloroform (1:1, v/v) and silica gel to give 4.1 g (50.3%) of white crystalline solid, mp 69–70 °C. Anal. Calcd for $C_{13}H_{12}F_3NO_2$: C, 57.57; H, 4.46; N, 5.16. Found: C, 57.74; H, 4.53; N, 5.18.

1,5-Dihydro-5-hydroxy-4-methyl-1-[3-(trifluoromethyl)phenyl]pyrrol-2*H*-one (2). A solution of 2.45 g of 1,5-dihydro-5-methoxy-4-methyl-1-[3-(trifluoromethyl)phenyl]pyrrol-2*H*-one in 8 mL of glacial acetic acid and 1 mL of 6 N hydrochloric acid was allowed to stand at room temperature until crystallization occurred (~3 days). The white crystals were collected, washed with water, and dried; mp 150–152 °C. Anal. Calcd for $C_{12}H_{10}F_3NO_2$: C, 56.03; H, 3.89; N, 5.45. Found: C, 56.13; H, 3.84; N, 5.52.

3-Methyl-1-[3-(trifluoromethyl)phenyl]maleimide. To a solution of 11.2 g (0.1 mol) of methylmaleic anhydride in 50 mL of acetic acid was added 16.1 g (0.1 mol) of

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3-(trifluoromethyl)aniline. The mixture was refluxed for 0.5 h, and the solvent was removed by a rotary evaporator. The resulting solid was purified by column chromatography using hexane–methylene chloride (1:1, v/v) and silica gel; mp 51–52 °C. Anal. Calcd for $C_{12}H_8F_3NO_2$: C, 56.47; H, 3.13; N, 5.49. Found: C, 56.65; H, 3.01; N, 5.52.

2-Methyl-4-oxo-4-[[3-(trifluoromethyl)phenyl]amino]butanoic Acid (4) and 3-Methyl-4-oxo-4-[[3-(trifluoromethyl)phenyl]amino]butanoic Acid (5). To a solution of 5.7 g (0.05 mol) of methylsuccinic anhydride in 60 mL of tetrahydrofuran was added 8.05 g (0.05 mol) of 3-(trifluoromethyl)aniline. The mixture was heated under reflux for 2 h, cooled, and concentrated on a rotary evaporator. The residue was dissolved in 100 mL of methylene chloride, washed twice with 50 mL of 0.5 M HCl, dried over anhydrous sodium sulfate, and concentrated to yield 12.05 g of white solid. The solid was a mixture of **4** as major component and **5** as minor component; mp 111–116 °C. Anal. Calcd for $C_{12}H_{12}F_3NO_3$: C, 52.36; H, 4.36; N, 5.09. Found: C, 52.36; H, 4.33; N, 5.10.

Hydrolysis Rate Measurement. Buffered aqueous solutions containing 5 mg/L of flurochloridone were prepared; the Clark and Lubs' buffer solutions (Lange, 1961) at pH 5, 7, and 9 at a concentration of 10^{-3} M were used. The aqueous solubility of flurochloridone is 35.1 mg/L at 25 °C. Six-milliliter portions of each test solution were placed in test tubes sealed with Teflon-lined caps. The sample tubes were placed in water baths thermostated at 25 ± 0.5 and 40 ± 0.5 °C; the baths were covered so that the test solutions were maintained in the dark. The content of flurochloridone was monitored at various times by extraction of a sample with toluene, followed by gas chromatographic analysis. The average recovery for flurochloridone was 96%.

No appreciable hydrolysis occurred at pH 7 at 25 °C, so these conditions were chosen for the following photolysis tests described later.

Photolysis Rate Measurement. The photoreactor chamber used for the rate measurement contains a UV black light lamp (GE No. F40 BL) mounted vertically inside a quartz preparative-scale photoreactor. This assembly is essentially identical with that described by Crosby (1969). The average UV intensity measured with a Rantech Radiometer (Model No. SN003) was 1600 $\mu W/cm^2$ at the surface of the lamp. An electric blower located at the bottom of the photoreactor chamber circulates room-temperature air through an adjustable opening to keep the temperature of the reactor at 25 ± 1 °C.

A stock solution (2.0 L) containing 12.00 mg/L of flurochloridone was prepared by dissolving a known amount of the compound in sterilized pH 7.0 buffer solution. A portion of this solution (1.4 L) was used to fill the preparative-scale photoreactor. A flask containing the balance of the solution was completely wrapped with aluminum foil and placed inside the reactor to serve as dark control. At various time intervals, duplicate 3-mL portions of test solution and the dark control solution were withdrawn for determination of flurochloridone content as described above.

Product Studies. The general procedure followed was to carry out the hydrolytic or photolytic decomposition and then isolate and identify the products formed. Product identification followed the standard spectrometric and chromatographic procedures summarized below.

Spectral and Chromatographic Identification Methods. Fourier transform nuclear magnetic resonance (FT-NMR) spectral analyses were performed with a Varian Model XL-200 multinuclear spectrometer. Proton spectra

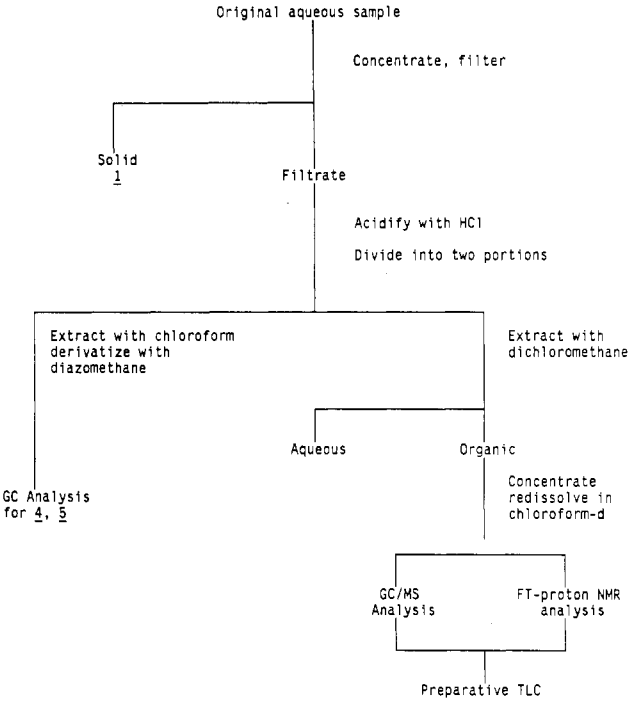

Figure 1. Isolation scheme for flurochloridone degradation products.

were obtained with a 5-mm H/F probe operated at a spectral width of 4 kHz, an acquisition time of 1 s, a pulse width of 10 μs (90°), and with 8K data points. Chemical shifts (ppm) are referenced to chloroform (δ 7.24). Coupling constants are expressed in hertz. Infrared (IR) spectra were obtained on a Beckman Model 4250 spectrophotometer.

A Finnigan Model 4021 gas chromatography–mass spectrometer was used for GC/MS analysis. The GC was equipped with a 25 m × 0.25 mm DB-17 fused silica capillary column. The column temperature was programmed from 40 to 230 °C at a rate of 30 °C/min and then to 280 °C at 8 °C/min. The mass spectrometer was operated in the electron-impact mode at an electron energy of 70 eV.

For thin-layer chromatography (TLC), E. Merck silica gel 60F-254 precoated glass plates (20 cm × 20 cm) were used. Visualizations of the TLC plates were made with a short-wave UV lamp (254 nm).

Gas chromatographic analyses were performed with a Hewlett-Packard Model 5880A chromatograph. For analysis of flurochloridone, the column used was an OV-101, 12 m × 0.20 mm, 0.33-μm film thickness, fused silica column coupled with nitrogen–phosphorus detector. The column was held at 90 °C for 1 min and then programmed to 220 °C at 20 °C/min. The retention time was 7.3 min. For analysis of the acidic component, the sample was derivatized to the corresponding methyl ester with diazomethane. The column used was a 15 m × 0.32 mm DB-5 fused silica capillary column coupled with a flame ionization detector; the column was held at 70 °C for 1 min and then programmed to 220 °C at 20 °C/min. The retention times of compounds **4** and **5** were 5.69 and 5.92 min.

Product Isolation and Identification. Large-scale reactions were carried out to facilitate product isolation. For hydrolysis, 2 L of test solution containing 24 mg/L of **1** in pH 9 buffer was maintained at 40 °C for 66 days and then analyzed. For photolysis, 3 L of test solution containing 30 mg/L of **1** in pH 7 buffer was prepared. The entire solution was used to fill two preparative-scale photoreactors. Both reactors were irradiated at 25 ± 3 °C

Table I. Spectroscopic Data of Hydrolysis and Photolysis Products of Fluorochloridone

compd	^{1}H NMR chem shift,[a] ppm (J, Hz)	MS data (m/z)[b]	IR,[c] cm^{-1}
2	2.11 (dd, CH_3, 3 H), 3.7 (br, OH), 5.71 (br d, methine), 5.79 (dq, vinyl), 7.35–7.50 (m, aromatic)	257 (M^+, 100), 238 (8), 228 (15), 212 (27), 69 (90)	3200 (CH), 1675 (C=O)
3	4.46 (br s, NCH_2), 4.61 (br s, OCH_2), 6.16 (br m, vinyl), 7.27–7.52 (m, aromatic, 2 H), 7.90 (br s, aromatic, 1 H), 8.04 (br d, aromatic, 1 H)	257 (M^+, 100), 226 (58), 198 (15), 172 (28), 145 (55)	
4, 5	1.32 (d, CH_3 in 4), 1.31 (d, CH_3 in 5), 2.42–3.08 (m, 3 H), 7.26–7.8 (m, aromatic)	275 (M^+, 7), 257 (4), 161 (100), 145 (8), 115 (15), 87 (17)	3300 (NH), 3500–2500 (OH), 1705 (C=O)
6	2.66, 2.76 (AB q, J = 17.1 nonequiv CH_2C=O), 3.77 (br s, NCH_2), 3.81, 3.99 (AB q, J = 10.6, nonequiv CH_2OH), 7.34–7.48, 7.78–7.88 (m, aromatic)	419[d] (M^+, 4), 404 (6), 329 (26), 316 (72), 73 (100)	
7[e]	2.80 (m), 3.06 (m) (methine attached to CH_2Cl), 2.85, 3.05 (br s, OH), 3.46–3.90 (m, CH_2Cl, CH_2N), 4.35 (d, J = 9.8, *trans* methine attached to OH), 4.56 (d, J = 6.3, *cis* methine attached to OH), 7.45–7.51 (m, aromatic)	293 (M^+, 25), 274 (4), 174 (100), 161 (30), 145 (23)	
8		275 (M^+, 56), 240 (100), 172 (56), 145 (65)	

[a] ^{1}H NMR spectra were obtained in $CDCl_3$ unless otherwise stated; s, t, m, d, dt, and br signify singlet, triplet, multiplet, doublet of doublet, doublet of triplet, and broad, respectively. [b] Mass spectra were obtained by EI at 70 eV, relative intensities enclosed in parentheses after m/z values. [c] Infrared spectra were obtained as KBr pellets. [d] Mass spectrum was obtained on the TMS derivative. [e] To confirm the positions of the hydroxy groups and the chlorine atom, an NMR spectrum run in DMSO-d_6 was obtained. The methine region centered at δ 3.91 and 4.15 exhibited doublet of doublets, suggesting additional coupling to hydroxyl proton (J = 9.7, 6.4 Hz; J = 7.4, 5.8 Hz). The hydroxy protons appeared at δ 5.80 and 5.87, both as doublets (J = 6.4, 5.8 Hz), due to coupling to the methine proton.

for 15.5 days, after which the test solutions were analyzed.

The final solutions were fractionated according to the scheme shown in Figure 1. After preliminary proton NMR and GC/MS analysis, the test solutions of the isolated fractions were concentrated for preparative TLC separation and then separated with use of a dichloromethane–ethyl acetate–acetic acid (40:59:1, v/v) solvent system with a silica gel TLC plate. After UV visualization the bands were scraped from the plates. The separated components were extracted from the silica gel with a chloroform–methanol mixture (1:1, v/v). The extracts were evaporated to dryness under nitrogen, and the compounds were redissolved in deuteriated chloroform for NMR and GC/MS analyses. Structural assignments were made on the basis of proton NMR chemical shifts, spin–spin coupling patterns, and mass spectral characteristics. Some of the putative products were synthesized. Confirmation was made by comparison of their spectral data and R_f values on TLC. Structures of the synthetic reference compounds were established by proton and carbon-13 NMR, infrared, and mass spectral data. The spectral data of the hydrolysis products and photoproducts are listed in Table I.

Product Quantitation by TLC and Autoradiography. Separate smaller scale tests using radiolabeled 1 were carried out to yield material balance information. For hydrolysis study, the test solution was prepared by pipetting 50 μL of the stock solution of the labled 1 in chloroform into a 10-mL culture tube equipped with a Teflon-lined screw cap. The chloroform was removed by evaporation under a stream of dry nitrogen. Sterilized pH 9 buffer solution (3.0 mL) was added to bring the radioactive material into solution. The culture tube was sealed and placed in a covered 40 °C bath for 32 days. For photolysis study, the test solution was prepared in the fashion using a pH 7 buffer solution and a quartz test tube fitted with a ground-glass stopper. The solution was irradiated for 7.5 days. For quantitation of the products, a 300-μL aliquot of the test solution was applied as a 2-cm strip on a 20 × 20 cm TLC plate. Previously identified components were cospotted for UV fluorescence viewing and comparison with radioactive components. After elution, the plate was air-dried, marked with a radioactive ink, and placed under X-ray film (Kodak XAR-5). Areas on the plates corresponding to the exposed areas on the films were scraped into 20-mL scintillation vials containing approximately 10 mL of scintillation fluid. The vials were agitated thoroughly and then placed in a Beckman Model LS-7500 liquid scintillation counter for determination of radioactivity.

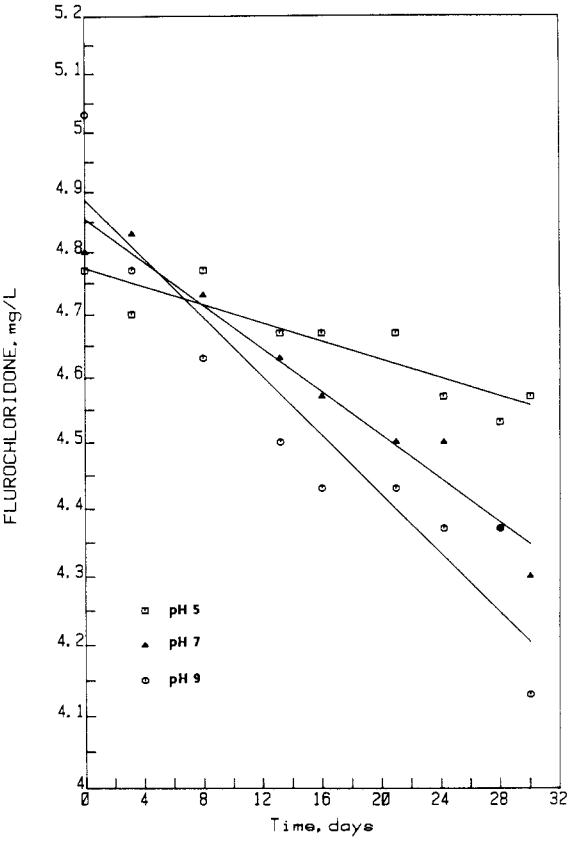

Figure 2. Semilogarithmic plot for hydrolysis rate of flurochloridone at 40 °C.

RESULTS AND DISCUSSION

Hydrolysis. The hydrolysis rate plot for flurochloridone is shown in Figure 2, where the y-axis is presented in logarithmic scale. Observable hydrolysis occurred only at pH 7 and 9 at 40 °C. The linearity observed for these conditions suggests that the hydrolysis reaction followed pseudo-first-order kinetics. The rate constants are 3.67×10^{-3} day^{-1} at pH 7 and 5.00×10^{-3} day^{-1} at pH 9. The hydrolytic half-lives are 190 days at pH 7 and 140 days at pH 9.

Table II. Distribution of Radiocarbon after Fluorochloridone Hydrolysis at pH 9 at 40 °C after 32 days

compd[a]	R_f[b]	radioact, dpm	rec,[c] %
1	0.756	397881	85.14
2	0.500	5406	1.14
4 + 5	0.348	5260	1.11
3	0.226	8388	1.78
6	0.096	3637	0.77
unknown	0.000	26814	5.73
total		447386	95.68

[a] Refer to Figure 3 for structures. [b] Solvent system: dichloromethane–ethyl acetate–acetic acid (40:59:1, v/v). [c] Calculated from initial count of 467 200 dpm and background count of 39 dpm.

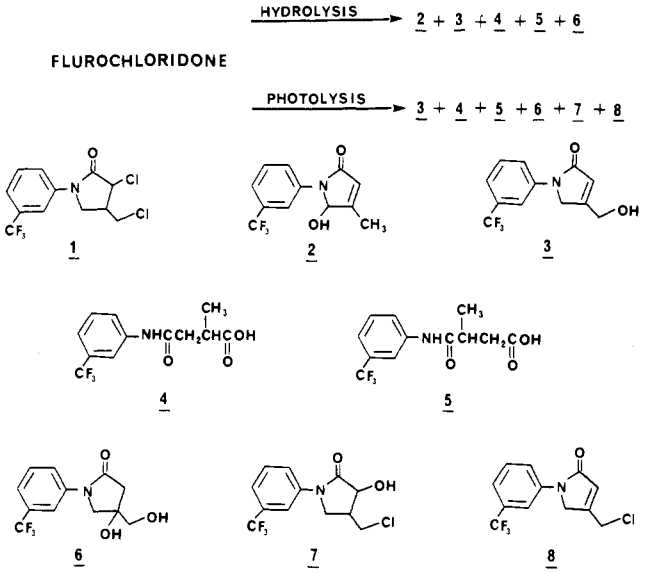

Figure 3. Hydrolysis and photolysis products of flurochloridone.

The product distribution for the hydrolysis of radiolabeled flurochloridone at pH 9 at 40 °C is summarized in Table II. About 96% of radioactivity was recovered; 85% of **1** remained unreacted. Five degradation products were isolated and identified. The structure of the most polar fraction, amounting to about 5.7% of the starting material, has not been identified. As shown in Figure 3, the five identified compounds, in decreasing order of concentration, are 1,5-dihydro-5-hydroxy-4-methyl-1-[3-(trifluoromethyl)phenyl]pyrrol-2*H*-one (**2**), 1,5-dihydro-4-(hydroxymethyl)-1-[3-(trifluoromethyl)phenyl]pyrrol-2*H*-one (**3**), 2-methyl-4-oxo-4-[[3-(trifluoromethyl)-phenyl]amino]butanoic acid (**4**), 3-methyl-4-oxo-4-[[3-(trifluoromethyl)phenyl]amino]butanoic acid (**5**), and 4-hydroxy-4-(hydroxymethyl)-1-[3-(trifluoromethyl)-phenyl]-2-pyrrolidinone (**6**).

Compound **2** was confirmed by comparison of its MS and NMR spectra with those of the synthesized authentic compound; the synthesis of **2** is outlined in Figure 4. The identity of compound **2** was further confirmed by oxidation to form 3-methyl-1-[3-(trifluoromethyl)phenyl]maleimide, which was shown to be identical with the product obtained by the reaction of 3-(trifluoromethyl)aniline with 2-methylmaleic anhydride. Compounds **4** and **5** were confirmed by comparison of their MS and NMR spectra with those of authentic compounds prepared by reaction of 3-(trifluoromethyl)aniline with methylsuccinic anhydride. The syntheses of **4** and **5** are also outlined in Figure 4. Authentic compounds of **3** and **6** were not available for comparison.

Formation of product **3** could be explained by dehydrochlorination at 3–4 position of the pyrrolidinone ring

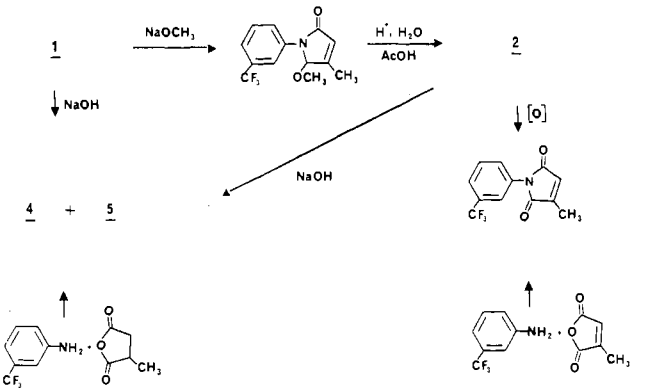

Figure 4. Synthesis scheme for some degradation products of flurochloridone.

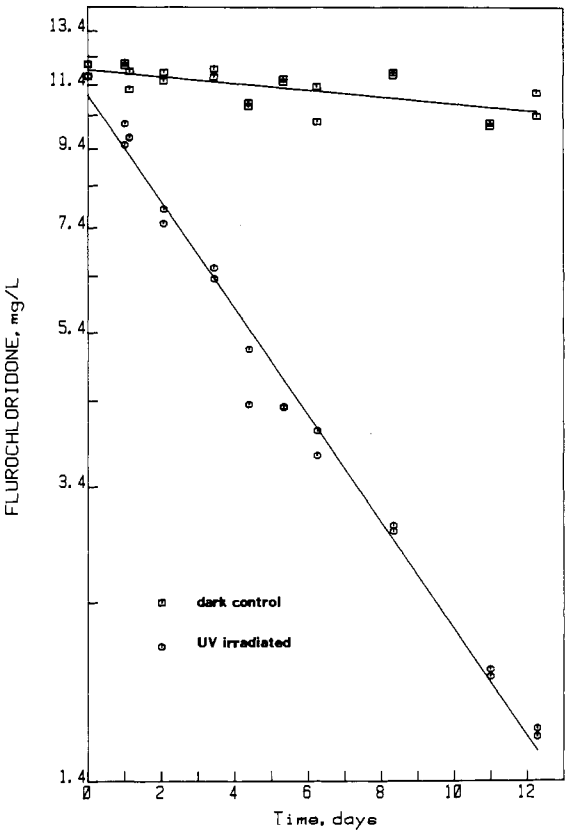

Figure 5. Photolysis rate plot for flurochloridone at pH 7 and 25 °C.

to form intermediate **8**, followed by hydrolysis of the chloromethyl group to hydroxymethyl group. Formation of product **2** is less obvious; the mechanism of this rearrangement is being investigated and will be reported in a separate paper. Products **4** and **5** could be formed by cleavage of N–C bonds in **2** by water. This cleavage reaction was confirmed by hydrolyzing an authentic sample of **2** to **4** and **5**. Hydration of the double bond in **3** could result product **6**.

Photolysis. The photolysis rate plots for flurochloridone are shown in Figure 5. The linearity of the plots again suggests the photolysis reaction follows pseudo-first-order kinetics. The rate constant is 0.161 day^{-1}, and the half-life is 4.3 days at pH 7 at 25 °C.

The product distribution for photolysis of radiolabeled flurochloridone is summarized in Table III. About 83% of radioactivity was recovered; approximately 20% was the starting material, **1**. Among the photodegradation products, six compounds have been identified as shown in

Table III. Distribution of Radiocarbon after Fluorochloridone Photolysis at pH 7 at 25 °C after 7.5 Days

compd[a]	R_f[b]	radioact, dpm	rec,[c] %
1	0.656	94967	19.55
8	0.640	14918	3.06
7 (trans + cis)	0.475	189588	39.03
unknown A	0.205	4765	0.97
3	0.188	27157	5.57
4 + 5	0.150	2898	0.59
6	0.088	10751	2.21
unknown B	0.000	58294	12.00
total		403338	82.98

[a] Refer to Figure 3 for structures. [b] Solvent system: dichloromethane–ethyl acetate (40:60, v/v). [c] Calculated from initial count of 485 528 dpm and background count of 39 dpm.

Figure 3 and two remain unidentified. Compounds 3–6 were identified as products of hydrolysis (above) and also occurred as photolysis products. The major photolytic degradation product, which contained 39% of the radioactivity, is 4-(chloromethyl)-3-hydroxy-1-[3-(trifluoromethyl)phenyl]-2-pyrrolidinone (7). Compound 7 is a mixture of cis and trans isomers. The position of the hydroxy group of 7 was established by observing spin–spin coupling between the hydroxy and the adjacent methine protons in the NMR spectrum run in DMSO-d_6. Compound 8, 4-(chloromethyl)-1,5-dihydro-1-[3-(trifluoromethyl)phenyl]pyrrol-2*H*-one was identified by GC/MS analysis. Authentic compounds of 7 and 8 were not available for comparison.

The formation of product 7 suggests the major photolytic pathway involves homolytic cleavage of the carbon–chlorine bond at the 3-position of the pyrrolidinone ring of flurochloridone. A homolytic C–Cl cleavage has been reported to account for the photodegradation products of propachlor (2-chloro-*N*-isopropylacetanilide) (Rejto et al., 1984). The free-radical intermediate can react with water to give 7 or eliminate a hydrogen radical to form 8.

ACKNOWLEDGMENT

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Registry No. 1, 61213-25-0; 2, 113321-91-8; 3, 113321-92-9; 4, 113321-93-0; 5, 113321-94-1; 6, 113321-95-2; 7, 113321-96-3; 8, 113321-97-4; 1,5-dihydro-5-methoxy-4-methyl-1-[3-(trifluoromethyl)phenyl]pyrrol-2*H*-one, 113321-98-5; 3-methyl-1-[3-(trifluoromethyl)phenyl]maleimide, 113321-99-6; methylmaleic anhydride, 616-02-4; 3-(trifluoromethyl)aniline, 98-16-8.

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Tetrachlorodibenzo-*p*-dioxin Residue Reduction through Cooking/Processing of Restructured Carp Fillets[1]

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Restructured carp fillets were used to study the potential of reducing dioxin levels during cooking. Carp from Saginaw Bay were mechanically deboned and washed. Samples were analyzed for 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) by selected ion monitoring GC–MS. Half was spiked to obtain levels of approximately 100 pptr. Fillets (7.5 cm in diameter) were roasted covered and uncovered at 177 °C to end internal temperatures of 60, 70, and 80 °C. To study the effect of surface area, fillets (10 cm in diameter) were roasted uncovered and charbroiled to 80 °C only. Fillets (7.5 cm in diameter) were also charbroiled to 60 and 80 °C. Spiked and unspiked (control) samples were cooked for each method/end temperature. Cooking resulted in reductions of TCDD in restructured carp surimi fillets. The magnitude of reduction was similar for unspiked and spiked fillets. Charbroiling resulted in greater TCDD reduction than roasting uncovered. Increasing internal end point temperature or surface area increased TCDD loss.

Bottom-feeding fish, i.e., carp and suckers, collected from Michigan rivers and streams in 1979 have been found to contain nondetectable to 120 pptr body weight of dioxins (Kaczmar, 1983). Fehringer et al. (1985) found fish collected in 1983 from Saginaw Bay and corresponding rivers to have the highest TCDD levels with values ranging from nondetectable to 102 pptr using HRGC-EC analyses. Fish from other Michigan rivers contained <10 pptr TCDD. Thus, every effort must be made to reduce the level of dioxins going into the environment and to reduce the levels of residues already in the environment. In preliminary studies using charbroiling of three carp fillets from Michigan rivers, extremely variable but substantial losses of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) ranging from 30 to 70% were recorded (Kaczmar, 1983). This

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