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## Perfluorinated Alkyl Carboxanilides: A New Class of Soil Insecticide

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EL-499, a mixture of 2'-bromo-4'-nitro-1,2,2,3,3,4,4,5,5,6,6-undecafluorocyclohexanecarboxanilide and isomeric (trifluoromethyl)perfluorocyclopentanecarboxanilides, represents a new chemical class of soil insecticide derived from electrochemical fluorination technology. The general structural requirement for the surprising insecticidal activity of the series of carboxanilides is  $\alpha$  branching in the perfluorinated alkyl moiety, which reduces hydrolytic lability relative to normal-chain acyclic derivatives.

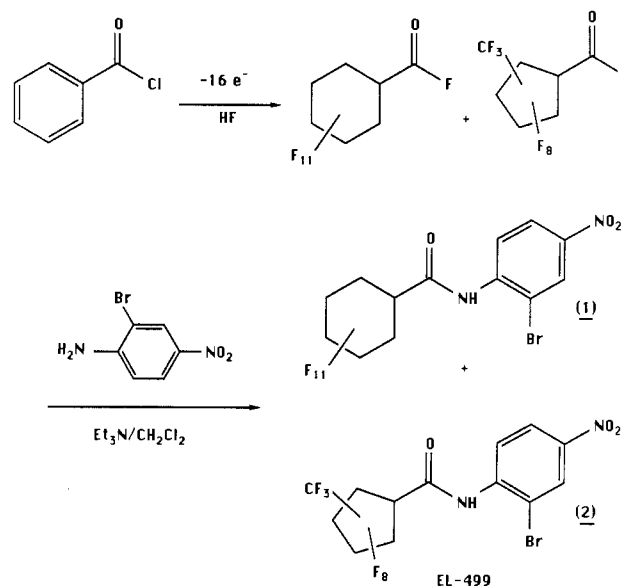
EL-499 (1, 2) is a novel soil insecticide candidate under investigation for the control of soil-borne insects including rootworm larvae in corn (Thompson et al., 1987). It is technically a mixture of isomeric six-carbon perfluorinated cycloalkyl carboxanilides prepared principally from perfluorocyclohexane carbonyl fluoride and 2-bromo-4-nitroaniline (Scheme I). Because of its excellent residual properties (Schultz et al., 1987) and low mammalian acute toxicity relative to many soil-applied phosphates and carbamates, EL-499, which is derived from electrochemical fluorination technology, represents an interesting new chemical class of insecticide. Indeed, these first-order derivatives of perfluorinated cycloalkyl carboxylic acids and acid halides, by virtue of their  $\alpha$  branching, exhibit properties differing dramatically from those of similar derivatives of the commercially available normal-chain perfluorinated carboxylic acid halides and anhydrides.

### EXPERIMENTAL SECTION

Insecticidal activity was determined by bioassays on southern corn rootworm (*Diabrotica undecimpunctata howardi*) larvae. The test compositions were solubilized in 50:50 acetone-ethanol and pipetted onto a dry, sandy loam soil on a w/w basis. Third instar larvae and corn seedlings were introduced after solvent evaporation with mortality readings taken 4 days post larval introduction.

Substituted anilines, benzoyl chlorides, and perfluorinated normal-chain carboxylic acid halides and anhydrides either were commercially available or were prepared by known methods. Carboxanilides were prepared from substituted anilines and acid halides in the presence of triethylamine and purified by either silica gel chromatography with toluene eluent or recrystallization from toluene or hexane to give satisfactory microanalyses (C, H, N) with yields, except as noted, of greater than 60%. All  $^{19}\text{F}$  NMR data were obtained on an IBM NR-80 at 75.26 MHz and are reported (ppm) relative to  $\text{CFCl}_3$  in chloroform solvent. Capillary GC/MS data were obtained on a Hewlett-Packard 5985B system with a 25-m SE-54 capillary column and temperature programming from 90

Scheme I. Synthesis of EL-499



to 260 °C at 20 °C/min. Relative isomer peak areas are reported without correction for detector response. Melting points were determined on a Mel-Temp and are uncorrected.

**EL-499 (1, 2).** The crude carboxanilide products derived from 3.2 kg (14.75 mol) of 2-bromo-4-nitroaniline, 8 kg (14.75 mol) of  $\text{C}_6\text{F}_{11}$  carbonyl fluoride mixture, and 2.1 kg (21 mol) of triethylamine in 20 L of ether were triturated with boiling hexane, leaving a sandy, insoluble residue. The hexane solution was evaporated to dryness and recrystallized from toluene to produce two crops: (5.5 kg (71%); combined mp 100–102 °C; capillary GC/MS relative peak area analysis, 90.4% cyclohexyl, 7.3% major (trifluoromethyl)cyclopentyl, 0.9% minor  $\text{C}_6\text{F}_{11}$ ).

**2'-Bromo-4'-nitro-1,2,2,3,3,4,4,5,5,6,6-undecafluorocyclohexanecarboxanilide (1).** Crude EL-499 (350 g) was recrystallized twice from 1.6 L of toluene and then once from 1.0 L of toluene. The resultant product (64 g) was chromatographed on 500 g of silica gel with toluene eluent. The product-containing fraction was

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evaporated to 250 cm<sup>3</sup> and allowed to crystallize in the refrigerator. The purified product was vacuum oven dried at 40 °C for 6 h: mp 108–109 °C; recovery, 37.5 g (10.7%); capillary GC relative peak area analysis, 98.3% cyclohexyl isomer; <sup>19</sup>F NMR, -117 (m), -121 (m), -124 (m), -130 (m), -133 (m), -137 (m), -139 (m), -141 (m), and -143 (m) [10 F], -180 (m, 1 F).

**2'-Bromo-4'-nitro-2,3,3,3-tetrafluoro-2-(trifluoromethyl)propionanilide (Olefin Process) (3).** Potassium fluoride (23 g, 0.4 mol), which was heated strongly with a Bunsen burner in a porcelain crucible and subsequently powdered, was added to 200 mL of a DMF solution of 2-bromo-4-nitrophenyl isocyanate (6 g, 0.025 mol). The mixture was placed in a 500-mL stainless-steel pressure vessel, purged with a small stream of hexafluoropropene, and heated to 70 °C while hexafluoropropene (25 g, 0.166 mol) was added from a preweighed supply cylinder at 10–20 psi. A pressure drop occurred as the gas reacted, and the remaining hexafluoropropene was added intermittently until the supply cylinder was empty. Heating was continued at 65–70 °C for 2.5 h, with pressure stabilized at 5 psi. The reaction vessel was cooled and the solution poured off and extracted with hexane. The DMF solution was poured into water and filtered. The solid products were taken up in chloroform, dried over sodium sulfate, filtered, evaporated, and chromatographed on silica gel with ethyl acetate/hexane (1:5). The front-running product was collected then rechromatographed on silica gel with gradient elution from 100% hexane to 100% ethyl acetate, yielding 1.65 g (16%) of a waxy solid: mp 57–9 °C; <sup>19</sup>F NMR, -74.5 (d, 6 F), -180.9 (m, 1 F).

**2'-Bromo-4'-nitro-2,3,3,3-tetrafluoro-2-(trifluoromethyl)propionanilide (Alkanoyl Fluoride Process, with Isomer Purification) (3).** 2-Bromo-4-nitroaniline (141 g, 0.65 mol) was dissolved in 3.5 L of diethyl ether. Triethylamine (71 g, 0.70 mol) was added all at once. A mixture of 2,3,3,3-tetrafluoro-2-(trifluoromethyl)propionyl fluoride and 2,2,3,3,4,4,4-heptafluorobutyryl fluoride (210 g, ~70% acid fluorides in fluorocarbon inerts, 0.70 mol) was bubbled in under a dry ice/acetone condenser. The reaction mixture was allowed to stir overnight at ambient temperature. It was then washed with cold, dilute HCl, and the organic layer was dried over MgSO<sub>4</sub> and evaporated to dryness. The residue and potassium carbonate (103 g, 0.75 mol) were stirred in 800 mL of 95% ethanol for 64 h at ambient temperature. The ethanol was evaporated, and the residue was extracted with water and then aqueous potassium carbonate three times. The aqueous extracts were filtered and acidified with concentrated HCl below 35 °C. The purified product was filtered and air-dried to produce 178 g (66%): mp 66 °C; GC peak height analysis, 99% branched isomer.

Compounds 4–7 were prepared from the corresponding perfluorinated alkanoyl halides.

**2'-Bromo-4'-nitro-2,3,3,3-tetrafluoro-2-(pentafluoroethoxy)propionanilide (4):** mp 49 °C; yield 25% following potassium carbonate/ethanol purification; <sup>19</sup>F NMR -77.3 (s, 3 F), -79.5 (d, 1 F, *J* = 152 Hz), -81.6 (s, 3 F), -84.1 (d, 1 F, *J* = 152 Hz), -125.3 (s, 1 F).

**2'-Bromo-4'-nitro-2,3,3,3-tetrafluoro-2-(heptafluoro-*n*-propoxy)propionanilide (5):** mp 49–51 °C; yield 35%; <sup>19</sup>F NMR -81.7 (s, 3 F), -82.2 (s, 3 F), -80.5 (d, 1 F, *J* = 150 Hz), -85.5 (d, 1 F, *J* = 150 Hz), -129.9 (s, 2 F), -133.2 (s, 1 F).

**2'-Bromo-4'-nitro-2,3,3,3-tetrafluoro-2-(octafluoromorpholino)propionanilide (6):** mp 50–52 °C; yield 74%; <sup>19</sup>F NMR -73.7 (m, 3 F), -82.7 (d, 2 F, *J* = 141 Hz), -88.3 (d, 2 F, *J* = 141 Hz), -86.9 (d, 2 F, *J* = 181 Hz), -92.6

(d, 2 F, *J* = 181 Hz), -134.9 (m, 1 F).

**2'-Bromo-4'-nitro-2-(trifluoromethyl)-2,3,3,4,4,5,5,5-octafluorovaleranilide (7):** mp 39–40 °C; yield 44%; <sup>19</sup>F NMR -72.5 (s, 3 F), -81.0 (s, 3 F), -116.7 (m, 2 F), -124.6 (m, 2 F), -179.7 (m, 1 F).

**2'-Bromo-4'-nitro-3-(trifluoromethyl)-1,2,2,3,4,4,5,5,6,6-decafluorocyclohexanecarboxanilide (8).** In a Teflon jar equipped with a stainless-steel condenser maintained from -40 to -50 °C, approximately 130 cm<sup>3</sup> of commercial anhydrous HF was subjected to a preelectrolysis to remove the last traces of water. An electrode pack of about 2-in.<sup>3</sup> size consisting of alternating nickel and carbon steel plates was used under a nitrogen atmosphere at a maximum current density of about 20 mA/cm<sup>2</sup> and at or below a cell voltage of 5.2 V relative to a copper reference electrode. Distilled *m*-(trifluoromethyl)benzoyl chloride (4.2 g, 0.02 mol) was added, and 5.6 amp h was passed (75% of theoretical). The reaction mixture was extracted with three 20-cm<sup>3</sup> portions of CFC<sub>3</sub>, and the extracts were added to 3.2 g of 2-bromo-4-nitroaniline (0.015 mol) and 2.5 g (0.025 mol) of triethylamine in 24 cm<sup>3</sup> of methylene chloride. The organic layer was washed with dilute HCl, dried over sodium sulfate, and chromatographed on silica gel with toluene to give 4.05 g (35%) from acid chloride: mp 55–60 °C; <sup>19</sup>F NMR -69.7 (s, 3 F), -115.4 to -137.6 (m, 8 F), -181.8 (s, 1 F), -186.2 (s, 1 F).

The following carboxanilides were prepared similarly:

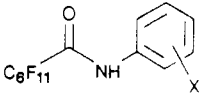
**2'-Bromo-4'-nitro-4-(trifluoromethoxy)-1,2,2,3,3,4,5,5,6,6-decafluorocyclohexanecarboxanilide (9):** mp 90–2 °C; yield 20% from *p*-(trifluoromethoxy)benzoyl chloride; <sup>19</sup>F NMR -53.8 (s, 3 F), -117.2 to -138.2 (m, 9 F), -179.4 (m, 1 F).

**2'-Bromo-4'-nitro-2-(trifluoromethyl)-1,2,3,3,4,4,5,5,6,6-decafluorocyclohexanecarboxanilide (10):** mp 66–80 °C (toluene); yield 5.1% from *o*-(trifluoromethyl)benzoyl chloride.

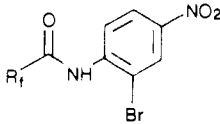
## RESULTS AND DISCUSSION

The key raw material for EL-499, a mixture of C<sub>6</sub>F<sub>11</sub> cycloalkyl carbonyl fluorides, is prepared from electrochemical fluorination of either cyclohexane carbonyl chloride (Kauck and Diesslin, 1951) or benzoyl chloride (Gambaretto et al., 1985) in about 50% cell yield via the Simon cell process in anhydrous HF. The principal cell product is perfluorocyclohexane carbonyl fluoride; however, ring contraction to smaller amounts of both 3- and 2-(trifluoromethyl)perfluorocyclopentyl carbonyl fluorides has been documented (Trollo and Gambaretto, 1971). Similarly, ring contraction can be confirmed from studies of the EL-499 anilide mixtures. Extensive purification of EL-499 produced 98% 2'-bromo-4'-nitro-1,2,2,3,3,4,4,5,5,6,6-undecafluorocyclohexanecarboxanilide (1). Its <sup>19</sup>F NMR spectrum was rich in difluoromethylene absorptions from -117 to -143 ppm, whereas the <sup>19</sup>F NMR spectrum of the EL-499 mixture exhibited minor trifluoromethyl absorptions from -73 to -81 ppm due to 2.

Unsubstituted and monosubstituted carboxanilides of perfluorinated cycloalkyl carboxylic acids (Fawcett et al., 1962; Murza and Chuchlova, 1979; Murza and Ismagilova, 1981; Diesslin et al., 1951, and 1952) and disubstituted carboxanilides of primary perfluorinated carboxylic acids (Bishop et al., 1964; DePasquale, 1976; Kirichenko et al., 1977; Kulikova et al., 1977; Fukui et al., 1958) are known, but the materials failed to produce soil insecticidal utility of commercial interest. Table I exhibits some aniline substitution patterns that produced acute toxicity to southern corn rootworm similar to terbufos. Most monosubstituted perfluorinated cyclohexyl carboxanilides did not control rootworm at soil application rates of up to 50×

**Table I. Effect of Aniline Ring Substitution on the Acute Larvicidal Activity of Cycloalkyl Carboxanilides**


X	southern corn rootworm <sup>a</sup> control, % (ppm)	mp, °C
2-Br	inactive (10.0)	94
4-NO <sub>2</sub>	50 (10.0)	166-169
2-Br, 4-NO <sub>2</sub>	100 (0.38-0.19)	100-102
2-Cl, 4-NO <sub>2</sub>	80 (0.75)	90-93
2-I, 4-NO <sub>2</sub>	100 (0.38)	125-127
2,3,4,5-Cl <sub>4</sub>	100 (0.75)	130
2,3,4,5,6-F <sub>5</sub>	100 (0.75)	135
2,5-Cl <sub>2</sub> , 4-NO <sub>2</sub>	100 (0.156)	99-100
terbufos	100 (0.19)	

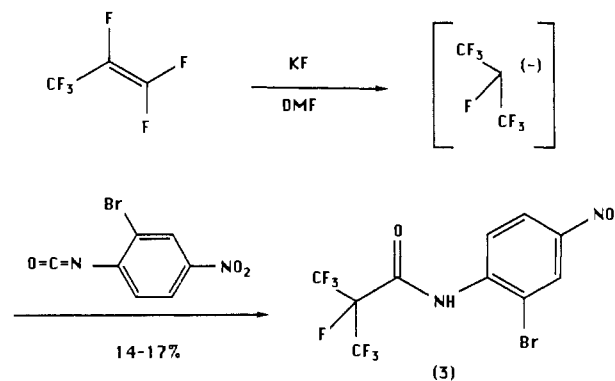
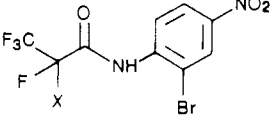
<sup>a</sup>Soil concentration (third instar).**Table II. Effect of  $\alpha$ -Branching on the Acute Activity of Acyclic Carboxanilides**



R <sub>f</sub>	southern corn rootworm control, % (ppm)	mp, °C
CF <sub>3</sub>	inactive (12.0)	107
CF <sub>3</sub> CF <sub>2</sub>	inactive (12.0)	107-108
CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub>	80 (6.0)	101
(CF <sub>3</sub> ) <sub>2</sub> CF (3)	100 (0.38-0.19)	66

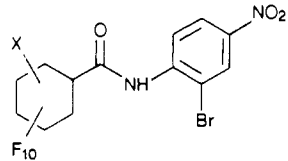
the rate of EL-499. Similarly, disubstituted carboxanilides of primary perfluorinated acids, such as trifluoroacetanilides, 2,2,3,3,3-pentafluoropropionanilides, and 2,2,3,3,4,4,4-heptafluorobutyranilides, did not produce activity comparable to that of EL-499 (Table II). In the latter cases, qualitative experiments with thin-layer chromatography indicated that the lower alkyl perfluoro carboxanilides were labile to base, being completely hydrolyzed to the acid and aniline with potassium carbonate/ethanol at room temperature after 3 days. In contrast, EL-499 remained essentially unchanged under the same conditions.

The sharp contrast in both lability and soil insecticidal activity observed above led to the speculation that hydrolysis is more effectively averted by the steric effects around the secondary acid function than by steric effects around the primary acid function, and the cyclohexyl molecule survives essentially intact in the soil, sustains lipophilicity, and exhibits its activity on corn rootworm. Further, similar steric effects might be expected from acyclic perfluorinated alkyl radicals containing branching  $\alpha$  to the carboxylic acid function, e.g. 2-(trifluoromethyl)-2,3,3,3-tetrafluoropropionanilides.

2'-Bromo-4'-nitro-2-(trifluoromethyl)-2,3,3,3-tetrafluoropropionanilide (3) was initially prepared by a novel heptafluoroisopropyl carbanion trapping reaction, albeit in only 16% yield, with 2-bromo-4-nitrophenyl isocyanate as described in Scheme II. Analogous reaction products have been derived from hexafluoropropene/cesium fluoride and carbon dioxide or carbonyl fluoride (Fawcett et al., 1962). The <sup>19</sup>F NMR spectrum of 3 indicated  $\alpha$ -branched isomeric purity with the trifluoromethyl absorption at -74.5 ppm and the methine fluorine absorption at -180.9 ppm. In potassium carbonate/ethanol, 3 exhibited similar resistance to hydrolysis and produced rootworm activity comparable to that of EL-499 (Table II).

**Scheme II. Haloolefin Synthesis of 3****Table III. Substituent Effects at the  $\alpha$ -Position of Acyclic Carboxanilides**


X	southern corn rootworm control, % (ppm)
CF <sub>3</sub> CF <sub>2</sub> O (4)	100 (0.75)
CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub> O (5)	50 (0.38)
 (6)	100 (0.38)
CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub> (7)	70 (0.38)

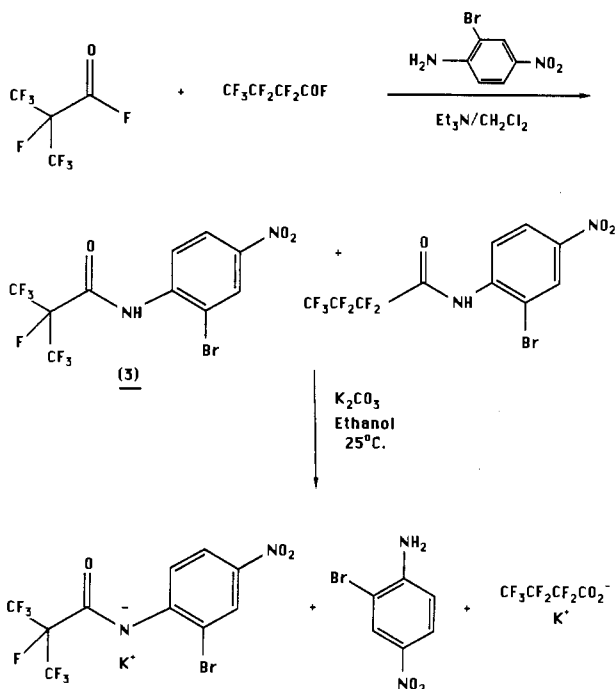
**Table IV. Effect of Substitution on the Acute Activity of Cycloalkyl Carboxanilides**


X	southern corn rootworm control, % (ppm)	X	southern corn rootworm control, % (ppm)
3-CF <sub>3</sub> (8)	60 (0.38)	2-CF <sub>3</sub> (10)	60 (0.38)
4-CF <sub>3</sub> O (9)	70 (0.38)		

Synthetic development efforts on the hexafluoropropene route failed to increase the reaction yields beyond 17%. However, a practical synthesis of 3 could be realized by exploiting the difference in reactivity of the primary and secondary perfluorinated carboxanilides. Reaction of a mixture of 2-(trifluoromethyl)-2,3,3,3-tetrafluoropropionyl fluoride and 2,2,3,3,4,4,4-heptafluorobutyryl fluoride (Gambaretto et al., 1985) with 2-bromo-4-nitroaniline gave an isomer mixture of carboxanilides, which was purified by selective hydrolysis of the primary carboxanilide in the presence of the secondary carboxanilide with potassium carbonate/ethanol (Scheme III). The water-soluble potassium salt of 3 was easily separated from the insoluble 2-bromo-4-nitroaniline byproduct, and 3 was regenerated with aqueous acid.

Alternate cyclic and acyclic perfluorinated alkyl radicals derived with electrochemical fluorination technology produced essentially similar acute activity to southern corn rootworm larvae as EL-499 (Tables III and IV). Clearly, the variety of perfluorinated alkyl radicals attests to simplicity of the lipophilic structural requirement for acute activity, which supports the hypothesis that steric effects

## Scheme III. Acid Halide Synthesis of 3 with Isomer Purification



via  $\alpha$ -branching averts hydrolysis and permits the exhibitions of insecticidal activity. The requirements for di-, tri-, tetra-, and pentasubstitution in the aniline ring are unique and account for the dramatic increase in soil insecticidal activity as compared with unsubstituted and monosubstituted carboxanilides previously prepared from similar  $\alpha$ -branched fluorinated alkyl carbonyl fluorides (Knunyants et al., 1961; Dear et al., 1971).

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**Registry No.** 1, 107349-85-9; 3, 105923-30-6; 4, 105923-75-9; 5, 105923-78-2; 6, 105923-77-1; 7, 105923-79-3; 8, 107803-97-4; 9, 107803-98-5; 10, 107351-23-5; EL-499, 111237-02-6; 2-Br-4-NO<sub>2</sub>-1-NCOC<sub>6</sub>H<sub>3</sub>, 105923-33-9; 2-Br-4-NO<sub>2</sub>-1-NH<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 13296-94-1; FCOC(F)(CF<sub>3</sub>)<sub>2</sub>, 677-84-9; CF<sub>3</sub>(CF<sub>2</sub>)<sub>2</sub>COF, 335-42-2; *n*-CF<sub>3</sub>C<sub>6</sub>H<sub>4</sub>COCl, 2251-65-2; *p*-CF<sub>3</sub>OC<sub>6</sub>H<sub>4</sub>COCl, 36823-88-8; *o*-CF<sub>3</sub>C<sub>6</sub>H<sub>4</sub>COCl, 312-94-7; 2'-bromoperfluorocyclohexanecarboxanilide, 111189-64-1; 4'-nitroperfluorocyclohexanecarboxanilide, 107349-86-0; 2'-chloro-4'-nitroperfluorocyclohexanecarboxanilide, 107349-90-6; 2'-iodo-4'-nitroperfluorocyclohexanecarboxanilide, 107349-87-1; 2',3',4',5'-tetrachloroperfluorocyclohexanecarboxanilide, 107350-42-5; perfluorocyclohexanecarboxanilide, 107350-45-8; 2',5'-dichloro-4'-nitroperfluorocyclohexanecarboxanilide, 107352-32-9; terbufos, 13071-79-9; 2'-bromo-4'-nitrotrifluoroacetanilide, 111189-65-2; 2'-bromo-4'-nitropentafluoropropionanilide, 111189-66-3; 2'-bromo-4'-nitroheptafluorobutyranilide, 111189-67-4.

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