

metals and, second, to be compatible with commercially available CRA workheads and power supplies with a minimum of hardware changes. The maximum volatilization temperatures achievable with them are too low for the determination of refractory analytes so the standard atomizer configurations must be used for metals less volatile than copper.

The principle followed in the design of these CRA atomizers is similar to that used by Littlejohn and Ottaway in their paper describing a Massmann tube furnace optimized for atomic emission analyses (17). Presumably then their furnace would evince lessened gas-phase matrix effects when used for absorbance measurements and, conversely, the modified CRA atomizers should be superior to the standard components for the atomic emission analysis of relatively volatile metals.

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Synthesis of the 38 Tetrachlorodibenzofuran Isomers and Identification by Capillary Column Gas Chromatography/Mass Spectrometry

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The 38 positional isomers of tetrachlorodibenzofuran have been synthesized by pyrolysis of specific polychlorinated biphenyl congeners, ultraviolet photolysis of pentachlorodibenzofurans, and chlorination of trichlorodibenzofurans by aromatic substitution. The specificity of these reactions in combination with capillary column gas chromatography with mass spectrometric detection has allowed each of these isomers to be identified based on their relative elution order.

In recent years the polychlorinated dibenzofurans [PCDFs] have been the subject of an intense research effort owing to their structural similarity to the polychlorinated dibenzo-p-dioxins [PCDDs] (1). Particular attention has been given to the tetrachlorodibenzofurans [TCDFs] for which there are 38 positional isomers, yet no analytical scheme allowing identification of specific TCDF isomers has yet been reported. In this paper we report on the synthesis of the 38 TCDF isomers by oxidative pyrolysis, under carefully controlled reaction conditions, of specific PCB congeners (2, 3), ultraviolet [UV] photolysis of pentachlorodibenzofurans [PenCDF], and chlorination by electrophilic aromatic substitution of specific trichlorodibenzofuran [TrCDF] isomers. Characterization of the TCDF isomers was accomplished by high-resolution gas chromatography/mass spectrometry and UV photolysis (4). The final result has been the development of an analytical scheme allowing for the analysis of 2378-TCDF (notation for symbols excludes the commas necessary in full names).

EXPERIMENTAL SECTION

Caution. Persons attempting to synthesize these compounds should first familiarize themselves with their safe handling and disposal.

PCB Congeners. All the PCB congeners used in this study were obtained from Ultra Scientific, Inc., Hope, RI, with the exception of 2,2',3,4'- and 2,3,3',4'-tetrachlorobiphenyl, 2,2',3,3',6-pentachlorobiphenyl, and 2,2',3,3',4,6'- and 2,2',3,3',5,6'-hexachlorobiphenyl which were received from C. A. Wachtmeister, Stockholms Universitet, Wallenberglaboratoriet S-10691 Stockholm, 2,3,4,4'-tetrachlorobiphenyl and 2,3,3',4,5-pentachlorobiphenyl which were received from J. Pyle, Miami University, Oxford, OH, and 2,2',3,4',5,6-hexachlorobiphenyl which was received from M. D. Mullins, EPA, Grosse Ile, MI.

The purity of the PCB congeners was generally greater than 97% assuming an equivalent FID response for all PCBs. In those cases where a given PCB congener was contaminated with another PCB congener, pyrolysis often yielded small amounts of PCDF isomers other than those expected. In all cases these were readily discernible from the desired PCDF isomer.

PCB Pyrolysis. Miniglass ampules 5-6 cm in length were prepared by sealing the large end of disposable borosilicate glass pasteur pipets (Model 13-678-20C, Fisher Scientific Co., Cincinnati, OH). Ten microliters of a solution of the PCB in hexane [10 µg/mL] was placed in the ampule and the solvent allowed to evaporate. The tip of the ampule was then flame sealed and the ampule placed in a large vial along with a cold junction referenced chromel-alumel thermocouple which was connected to a digital voltmeter to allow accurate temperature measurements. The ampule and thermocouple were placed into a muffle furnace [Type 1500, Thermolyne Corp., Dubuque, IA] operated at 600 °C. When the temperature of the ampule reached 550 °C, pyrolysis was allowed to continue for an additional 5 s. The ampule was removed from the furnace and allowed to cool to ambient temperature at which time it was opened and the contents thoroughly rinsed out with 1 mL of hexane.

In some cases unreacted PCBs were removed by subjecting the pyrolysate to chromatographic separation on a minicolumn of Woelm Basic Alumina (ICN Pharmaceuticals, Cleveland, OH) by eluting the PCBs with 10 mL of 2% methylene chloride in hexane, and then eluting the retained PCDFs with 15 mL of 50%

Table I. Synthesis Routes for the 38 TCDFs

TCDF	primary PCB congener	secondary PCB congener	PCDF dechlorinated	PCDF chlorinated	confirmed by photolysis
1234	23456, ^a 2345 ^a	22'346			
1236	22'346		12367		
1237	2344' ^a	22'344'6	12367	123	
1238			12348	238, 123, 128	yes
1239	22'346			123	
1246	22'33'6	22'356		124	
1247	22'34'56 ^a			124	
1248	22'355'6 ^{a,d}			124, 128	yes ^b
1249	22'356			124	
1267	233'4'	22'33'46'	12367		
1268	22'33'56'		12468		
1269	22'3'5 ^a	22'33'6, 22'33'66'			
1278	233'4'		23469	238, 128	yes ^b
1279	22'34' ^{a,c}	22'34' ^c			
1289	22'33'	22'33'6, 22'33'66'			
1346	22'346		13467		
1347	22'344'6	22'44'6	13467		
1348	22'455'	22'45'6			
1349	22'466'	22'346			
1367	23'44'	22'44'6, 23'44'6	13467, 12367		
1368	23'45'6 ^a	22'45'6			
1369	22'4'56	22'466', 22'45'6			
1378	22'44'5'6 ^a	23'44', 23'44'6			
1379	22'44' ^a	22'44'6			
1467	23'4'5	22'33'46'	13467, 23469		
1468	23'55' ^a	22'33'56'	23469		yes ^b
1469	22'55' ^a	22'33'66'			
1478	23'4'5'	22'455'	23469		
2346	22'33'45	233'45			
2347	22'344'5 ^a				
2348	22'3455' ^a	22'455', 233'45		238	
2349	22'3456' ^a				
2367	33'44'		12367		
2368	22'455'		23468	238	yes ^b
2378	22'44'55' ^a	33'44'			yes
2467	<i>d</i>		23468		yes ^b
2468	<i>d</i>		23468		yes ^b
3467	22'33'44' ^a	33'44'	13467		

^a Represents those cases in which a single TCDF isomer was formed from the pyrolyses. ^b These TCDFs were also received from D. Firestone and checked against our own syntheses. ^c These are identical PCB congeners from different sources. Pyrolyses of both gave identical products. ^d The relative retention times of these isomers were identical with the same isomer synthesized by the palladium acetate cyclization of the appropriate diphenyl ethers (6, 7).

methylene chloride in hexane. In many instances PCDF isomers were separated from one another by using the HPLC techniques described below.

Reversed-Phase High-Performance Liquid Chromatography (RP-HPLC). Separation of isomers was carried out with a 25 cm × 4.6 mm μ Bondapak C₁₈ column (Waters Associates, Inc., Milford, MA) with 75% acetonitrile/25% water as the eluent at a flow rate of 0.75 mL/min. Altex Model 110 pumps (Beckman Instruments, Inc., Fullerton, CA) were used with a Valco Model CV-6-UHPA-N60 injector (Valco Instruments Co., Houston, TX). Fractions were collected on a Gilson FC-80 fraction collector (Gilson Medical Electronics, Middleton, WI) with sample detection being carried out with a Beckman Model 100-10 UV spectrophotometer operated at 235 nm.

Normal-Phase (NH₂) High-Performance Liquid Chromatography (NH₂-HPLC). The normal-phase HPLC system used was the same as that described for reverse-phase HPLC with the exception that a 25 cm × 4.6 mm Zorbax NH₂ column [Du Pont, Analytical Instrument Division, Wilmington, DE] was used with hexane as the eluent at a flow rate of 1.5 mL/min.

Gas Chromatography/Mass Spectrometry (GC/MS) Analyses. GC/MS analyses were performed with a Hewlett-Packard 5985B quadrupole GC/MS system operated in the electron impact ionization mode. capillary columns that were used included a 60 m × 0.25 mm i.d. glass coated with SP-2330 (Supelco, Bellefonte, PA) and a 30 m × 0.25 mm i.d. fused silica coated with SE-54 (J & W Scientific, Rancho Cordova, CA). The capillary columns were directly coupled to the ion source via a 45 cm × 0.20 mm i.d. length of fused silica coated with SE-30. Column conditions for the SP-2330 column were as follows: 200 °C, 1 min

isothermal, 8 °C/min to 250 °C, isothermal at 250 °C for the duration of the run time; hydrogen carrier gas at 15 psi yielding a linear flow velocity of 40 cm/s. For the SE-54 column the conditions were as follows: 200 °C, 1 min isothermal, 10 °C/min to 275 °C, isothermal at 275 °C for the duration of the run time; helium carrier gas at 7 psi yielding a linear flow velocity of 38.5 cm/s. All samples were introduced via splitless injection with the injector at 275 °C using tetradecane as the solvent.

Mass fragmentograms were obtained by monitoring ions characteristic of each class of PCDF possible in a synthesis experiment. This included the molecular ions for the dichloro- and trichlorodibenzofurans, the molecular ions as well as the highly characteristic COCl loss for the tetra- and pentachlorinated dibenzofurans. In addition, ions characteristic of polychlorinated diphenyl ethers were also monitored to ensure that these compounds did not interfere in the analyses. An internal standard, [³⁷Cl]₂2378-TCDF (KOR Isotopes, Cambridge, MA) was coinjected with each sample, requiring that *m/z* 312 also be monitored during each run.

Spectral Sample Isolation. In certain cases it was necessary to obtain significant amounts of PCDFs (10–50 μ g) for further chlorination or photolysis studies. These PCDFs were obtained by the RP-HPLC separation of a mixture of PCDFs formed by the extensive chlorination of dibenzofuran (5). In many instances the RP-HPLC fractions were further processed by NH₂-HPLC to isolate a single PCDF isomer. The identity of this isomer was then determined by its GC characteristics on the two capillary columns used in these analyses.

Chlorination. Chlorination of the PCDFs was accomplished by placing a solution of 200 μ L of CCl₄ containing the PCDF to

Table III. Cross Correlation Chart of TrCDF Isomers vs. TCDF Isomers

TCDF	TrCDF																												
	123	124	126	127	128	129	134	136	137	138	139	146	147	148	149	234	236	237	238	239	246	247	248	249	346	347	348	349	
1234	•	•					•									•													
1236	•		•					•									•												
1237	•			•					•									•											
1238	•				•					•									•										
1239	•					•					•									•									
1246		•	•									•									•								
1247		•		•									•									•							
1248		•			•									•									•						
1249		•				•									•									•					
2346																•	•				•				•				
2347																•		•				•				•			
2348																•			•				•				•		
2349																•				•				•			•		
1346							•	•					•												•				
1347							•		•				•													•			
1348							•			•				•												•			
1349							•				•				•											•			
1267			•	•																							•	•	
1268			•		•																			•	•				
1269			•			•								•	•														
1278				•	•															•	•								
1279				•		•				•	•																		
1289				•	•																								
1367								•	•																	•		•	
1368								•		•												•			•				
1369								•			•			•															
1378									•	•									•		•								
1379									•		•									•									
1467												•	•												•			•	
1468												•		•								•			•				
1469												•			•														
1478													•	•							•								
2367																	•	•								•	•		
2368																	•		•			•	•						
2378																		•	•										
2467																					•	•			•		•		
2468																					•		•						
3467																						•			•	•			

of TCDFs. In order to identify a given TCDF, it was necessary to perform a pyrolysis of another PCB congener that would yield the desired TCDF among others. An assignment of the particular TCDF was made based on the match of the chromatographic retention characteristics of the various pyrolysates (as shown in Figure 2). As an example of this process, consider the pyrolysis of 3,3',4,4'-tetrachlorobiphenyl yielding 3467-, 2378-, and 2367-TCDF. By examination of Table I and Figure 2 it is seen that 2378-TCDF is the only TCDF formed from the pyrolysis of 2,2',4,4',5,5'-hexachlorobiphenyl and 3467-TCDF is the only TCDF formed from the pyrolysis of

2,2',3,3',4,4'-hexachlorobiphenyl. Therefore the remaining TCDF must be 2367-TCDF. This identification was subsequently confirmed by the dechlorination of 12367-PenCDF to form 2367-TCDF in a mixture with 1236-, 1237-, 1267-, and 1367-TCDF.

It was found to be convenient to prepare a cross correlation chart (Table II) which lists the PCB congeners on one axis and the possible TCDF isomers formed by pyrolysis of these PCB congeners on the other axis. This chart shows the possible PCB congeners used in this study which may be pyrolyzed to yield a given TCDF or, conversely, which TCDF

Table IV. Cross Correlation Chart of PenCDF Isomers vs. TCDF Isomers

TCDF	PenCDF																												
	12346	12347	12348	12349	12467	12468	12469	12478	12479	12367	12368	12369	12378	12379	12389	12489	13467	13468	13469	13478	13479	13489	23467	23468	23469	23478	23479	12678	
1234	•	•	•	•																									
1236	•									•	•	•																	
1237		•								•			•	•															
1238			•								•		•		•														
1239				•								•			•	•													
1246	•				•	•	•																						
1247		•			•			•	•																				
1248			•			•		•									•												
1249				•			•		•							•													
2346	•																						•	•	•				
2347		•																					•				•	•	
2348			•																					•			•	•	
2349				•																					•			•	•
1346	•																•	•	•										
1347		•															•			•	•								
1348			•															•		•	•								
1349				•															•		•	•							
1267					•					•													•						•
1268						•					•					•													•
1269							•					•				•							•						
1278								•						•		•													•
1279									•						•	•							•						
1289															•	•													
1367										•							•					•							•
1368										•	•						•												•
1369									•			•						•			•								•
1378													•	•							•								•
1379														•								•							
1467					•												•			•						•			
1468						•												•						•	•				
1469							•												•		•								
1478								•				•									•					•			
2367										•											•		•				•		
2368									•		•													•			•		
2378													•														•		
2467					•													•					•	•					
2468						•																		•	•				
3467																	•						•						

(or TCDF mixtures) would be formed from the pyrolysis of a given PCB congener.

Unless the primary PCB pyrolysis yielded a single TCDF isomer, it was necessary to prepare a confirming TCDF isomer. If the PCB congener required for a second pyrolysis was not available, either chlorination of an appropriate TrCDF or photolytic dechlorination of an appropriate PenCDF was used to give the needed TCDF. Cross correlation charts were prepared illustrating the TCDFs which may be formed from chlorinating a given TrCDF (Table III) and also a cross chart

showing the TCDFs which may be formed from dechlorinating a given PenCDF (Table IV). In Table III all the TrCDFs are listed on one axis and all the TCDFs are listed on the other axis. This chart may be used both for determining all the TCDFs that may result from the chlorination of a given TrCDF isomer and conversely for determining all the possible TrCDFs that may form by the UV photolysis of a given TCDF. The same principle is applicable for Table IV which gives TCDFs vs. PenCDFs. Obviously the tri- and pentachlorodibenzofurans used in the chlorination and dechlorination

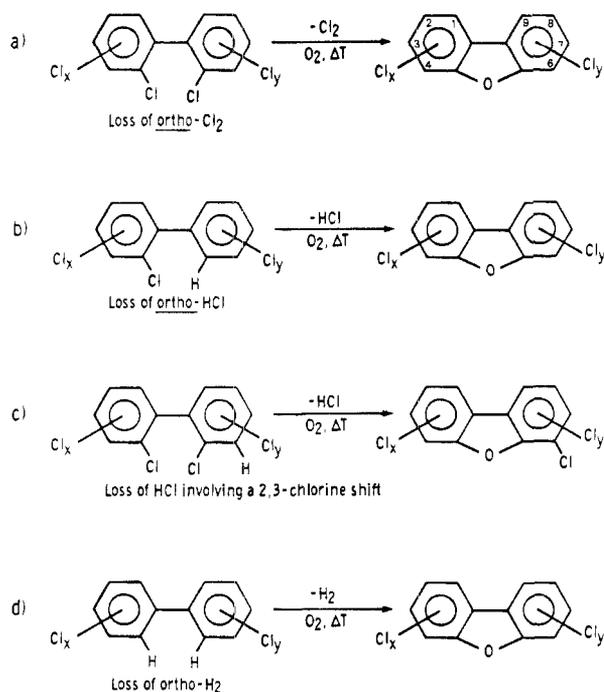


Figure 1. The four reaction routes from which PCDFs are produced from the pyrolysis of PCB congeners.

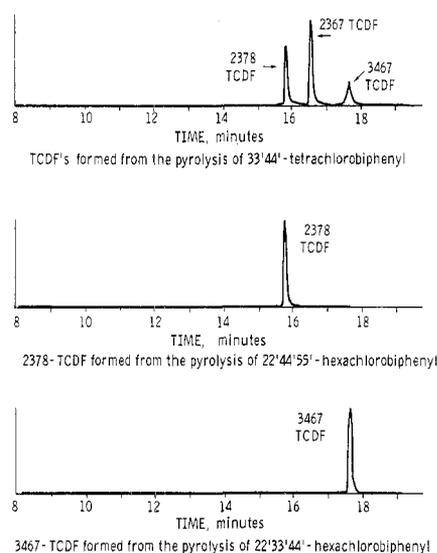


Figure 2. The GC/MS analysis of the TCDFs formed from the pyrolysis of various PCB congeners (SP-2330 capillary column).

Table V. Synthesis Routes for the TrCDF Isomers Used in This Study

TrCDF isomer	primary PCB congener	secondary PCB congener	dechlorination
123	234 ^a	22'346	
124	2356 ^a	22'356	
128	22'3'5		1278
128	22'455'	23'4'5	

^a Represents those cases in which a single TrCDF isomer was formed from the pyrolysis.

reactions, respectively, also had to be of confirmed validity. Table V lists the TrCDFs which were chlorinated in this study and their routes of synthesis and confirmation. Again those PCB congeners which when pyrolyzed gave a single TrCDF isomer are footnoted. The 128-TrCDF was confirmed by the photolytic dechlorination of 1278-TCDF. Table VI gives the

Table VI. Synthesis Routes for the PenCDF Isomers Used in This Study

PenCDF isomer	primary PCB congener	secondary PCB congener
12367	22'33'44' ^a	22'344'6; 22'33'44'6
12348	22'3455'6 ^a	233'45; 22'3455'
23469	22'3456'	22'3455'
13467	22'3455'	22'344'5'
23468	22'3455'	
23467	233'44'5	22'344'5

^a Represents those cases in which a single PenCDF isomer was formed from the pyrolysis.

Table VII. Relative Retention Times (RRT) for the 38 TCDFs Listed in Order of Increasing RRT on Both SP-2330 and SE-54 Capillary Columns

TCDF	RRT ^a (SP-2330)	TCDF	RRT ^a (SE-54)
1368	0.550	1368	0.846
1378	0.620	1468	0.870
1379	0.620	2468	0.882
1347	0.630	1347	0.895
1468	0.640	1247	0.897
1247	0.647	1367	0.900
1367	0.652	1378	0.900
1348	0.666	1346	0.902
1346	0.687	1246	0.903
1248	0.690	1348	0.909
1246	0.707	1379	0.909
1268	0.707	1248	0.915
1237	0.713	1268	0.925
1478	0.717	1478	0.929
1369	0.720	1467	0.935
2349	0.754	1237	0.943
1234	0.758	1369	0.944
2468	0.763	2368	0.947
1238	0.763	2467	0.961
1467	0.769	1238	0.962
1236	0.772	1469	0.966
1349	0.800	2349	0.966
1278	0.807	1234	0.968
1267	0.847	1278	0.979
1279	0.850	1267	0.991
1469	0.861	1349	0.991
1249	0.867	1249	1.000
1268	0.867	2378	1.000
2467	0.919	1279	1.002
2347	0.963	2346	1.004
1239	0.965	2347	1.004
1269	0.998	2348	1.004
2378	1.000	2367	1.026
2348	1.011	3467	1.037
2346	1.035	1269	1.038
2367	1.050	1239	1.051
3467	1.135	1236	1.051
1289	1.210	1289	1.103

^a All RRT based on [³⁷Cl₄]2378-TCDF which by definition = 1.000.

same information for the PenCDFs used in the synthesis of TCDFs. It should be reemphasized that when chlorination or dechlorination was performed as a synthetic route, it was necessary to isolate the starting PCDF from any other PCDFs. Upon chlorination for example, TrCDFs and TCDFs can be formed from dichlorodibenzofurans created during the pyrolysis of the original PCB congener, thus complicating the analysis. Isolation of the PCDF in these cases was effected by HPLC fraction collection as discussed earlier with the purity of the collected fraction being confirmed by GC/MS analysis.

GC Characteristics. The GC retention characteristics are listed for both the Sp-2330 and the SE-54 columns in Table

VII. These are relative retention times calculated relative to [$^{37}\text{Cl}_4$]2378-TCDF which is assigned a value of 1.000. Of the two columns, the SP-2330 is clearly superior for the resolution of these isomers; however, both columns serve as very useful tools and, when used together, provide the primary means of performing 2378-TCDF isomer specific analyses. There are one or more TCDF isomers coeluting with 2378-TCDF on both columns; however, due to the differences in the respective liquid phases, the coeluting isomers are resolvable on the alternate column. On SE-54, 2378-TCDF coelutes with seven other isomers using a retention time window of 1.000 ± 0.01 . On SP-2330 only 1269-TCDF elutes with 2378-TCDF. TCDF analyses were normally conducted on the SE-54 column first due to its ease of handling and higher temperature limit allowing a broader range of PCDFs to be analyzed. If a TCDF was found within the retention time window of 2378-TCDF, then any peaks eluting at relative retention time = 1.035 ± 0.01 were also quantitated since this window contains the 2367-, 3467-, and 1269-TCDFs. The analysis was repeated on SP-2330 and the peaks eluting in the 2378-TCDF window are quantitated. The now completely resolved 2367- and 3467-TCDFs were quantitated, and that sum was subtracted from the sum of 2367-, 3467-, and 1269-TCDFs on SE-54. This difference is the amount of 1269-TCDF present in the sample. This in turn is subtracted from the quantified peak in the 2378-TCDF window of SP-2330, the difference being the amount of 2378-TCDF present in the sample. As a further confirmatory step, a TCDF whose identity is in question may be isolated and subjected to UV photolysis as discussed elsewhere (4). The correct TrCDFs should then be formed as shown in Table IV and thus this technique serves as a valuable tool in TCDF isomer identification.

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Registry No. 1234-TCDF, 24478-72-6; 1236-TCDF, 83704-21-6; 1237-TCDF, 83704-22-7; 1238-TCDF, 62615-08-1; 1239-TCDF, 83704-23-8; 1246-TCDF, 71998-73-7; 1247-TCDF, 83719-40-8; 1248-TCDF, 64126-87-0; 1249-TCDF, 83704-24-9; 1267-TCDF, 83704-25-0; 1268-TCDF, 83710-07-0; 1269-TCDF, 70648-18-9; 1278-TCDF, 58802-20-3; 1279-TCDF, 83704-26-1; 1289-TCDF, 70648-22-5; 1346-TCDF, 83704-27-2; 1347-TCDF, 70648-16-7; 1348-TCDF, 64126-87-0; 1349-TCDF, 83704-28-3; 1367-TCDF, 57117-36-9; 1368-TCDF, 71998-72-6; 1369-TCDF, 83690-98-6; 1378-TCDF, 57117-35-8; 1379-TCDF, 64560-17-4; 1467-TCDF, 66794-59-0; 1468-TCDF, 82911-58-8; 1469-TCDF, 70648-19-0; 1478-TCDF, 83704-29-4; 2346-TCDF, 83704-30-7; 2347-TCDF, 83704-31-8; 2348-TCDF, 83704-32-9; 2349-TCDF, 83704-33-0; 2367-TCDF, 57117-39-2; 2368-TCDF, 57117-37-0; 2378-TCDF, 51207-31-9; 2467-TCDF, 57117-38-1; 2468-TCDF, 58802-19-0; 3467-TCDF, 57117-40-5; 123-TrCDF, 83636-47-9; 124-TrCDF, 24478-73-7; 128-TrCDF, 83704-34-1; 238-TrCDF, 57117-32-5; 12367-PenCDF, 57117-42-7; 12348-PenCDF, 67517-48-0; 23469-PenCDF, 83704-35-2; 13467-PenCDF, 83704-36-3; 23468-PenCDF,

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