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Supplementary Material Available. *An annotated listing of the computer program in Fortran, wind direction and speed data, and an additional figure will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material for the papers in this issue may be obtained from the Business Operations Office, Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036. Remit check or money order for $4.00 for photocopy or $2.50 for microfiche, referring to code number ES&T-76-1223.*

Product File

PCB and PCT Contamination of the Environment near Sites of Manufacture and Use

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■ A survey was conducted to assess the degree of environmental contamination with polychlorinated biphenyls (PCB's) and polychlorinated terphenyls (PCT's) in the vicinity of the single U.S. manufacturing facility in Sauget, Ill., and two users in the investment casting industry located in urban Chicago and suburban Detroit. PCB concentrations were found in soils as high as 18 ppm near the manufacturer and 5.2 ppm near one of the users. PCT's were detected as high as 13 ppm in the vicinity of one user facility. The distribution of PCB's and PCT's suggested an airborne mode of transport from the source. Analysis of wastewaters indicated the discharge of PCB's and PCT's in the μg/l. level.

Polychlorinated biphenyls (PCB's) have a basic chemical structure as shown in Figure 1. From 1 to 10 chlorine atoms may be attached to the structure, resulting in 210 possible isomers. PCB's have certain physical properties, including high heat capacity, chemical stability, and excellent dielectric properties which make them highly desirable for a number of industrial uses. Concern has been expressed in recent years over the widespread dispersion of PCB's in the environment. These compounds are suspected to have significant adverse effects on organisms at the top of the food web, including man. Biomagnification of this contaminant in natural predator-prey food chains has been documented by numerous studies. To compliciate the problem, the rate of degradation of PCB's in the environment, particularly the higher substituted isomers, is apparently extremely slow. This situation allows for significant accumulation of these persistent chlorinated organic compounds in the environment. A thorough discussion of the problem and of the environmental data that are available has been presented by Selikoff (*1*) and the U.S. Government Interdepartmental Task Force (*2*).

Polychlorinated terphenyls (PCT's) have a similar but more limited industrial application. A generalized PCT structure is illustrated in Figure 1. There exists little information concerning PCT's in the environment. The environmental behavior of PCT's may be expected to be similar to that of PCB's.

The Monsanto Co. is the sole domestic producer of both PCB's and PCT's under the trade name of Aroclors. In response to the rising concern over PCB's in the environment, the Monsanto Co. has voluntarily taken two actions to reduce the environmental hazard. Beginning in 1970, the production of Aroclor was voluntarily reduced by the manufacturer, and supply was discontinued to those users who could not control release to the environment. Production of the more highly chlorine substituted Aroclor mixtures, including all PCT's, has since been curtailed or discontinued, and a lower substituted, refined mixture (Aroclor 1016) has been introduced. Some research indicates the lower substituted PCB isomers do not persist as long in the environment as the higher substituted isomers, and that they may be amenable to biological degradation, hence reducing the potential hazards to the environment. A recent public announcement by Monsanto Co. (January 1976) has indicated their intention to suspend all production of PCB's.

PCB's are presently employed in electrical capacitors, transformers, vacuum pumps, and gas-transmission turbines (*3*). Both PCB's and PCT's are currently used in the investment casting industry as a major constituent of the wax material used. This material is fashioned into the shape to be cast. A refractory mold is then formed around the wax shape. The wax is melted and poured from the mold, leaving the mold prepared for casting of the shape in metal.

The investment casting industry has not been supplied by Monsanto Co. for several years. PCB's and PCT's have been imported from foreign sources to meet this demand. Hutzinger et al. (*3*) list the origin and trade name of foreign manufactured PCB mixtures.

Sampling and Analysis

A field sampling program was conducted in early 1975 under the direction of the Environmental Protection Agency to assess the effectiveness of voluntary controls by industry in eliminating the release of PCB's to the environment. Samples were taken in the vicinity of the domestic manufacturing facility and two PCB and PCT user facilities. The production facility is located in Sauget, Ill., the user facilities are located in urban Chicago, Ill., and suburban Detroit, Mich.

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Both users surveyed are involved in the investment casting industry.

Surface soil samples were collected at each site from the plant boundary to a distance of approximately 2000 m in four directions, subject to the physical constraints encountered. At each soil sample point, five subsamples were collected within a radius of 10 m to a depth of 2.5 cm over a 100 cm^2 area. Vegetative matter was removed with forceps, and the five subsamples were composited and thoroughly mixed. This mode of sampling was designed to collect a homogeneous surface soil sample representative of the chosen sample point. Every attempt was made to select sample points where surface vegetation was sparse and where it appeared unlikely wind scour would be a major soil transport factor. Samples were placed in precleaned wide-mouth glass jars with aluminum foil-lined closures.

Water samples were taken in 1-L glass containers, composited, and stored in 4-L precleaned glass-stoppered reagent bottles. Soil samples were extracted in a soxhlet apparatus with 1:1 nanograde hexane/acetone, dried with sodium sulfate, and concentrated on a Kuderna-Danish apparatus. The procedure follows that prescribed by the EPA (*4*). Water samples were triple extracted with 15% nanograde ethyl ether in hexane, dried with sodium sulfate, and concentrated on a Kuderna-Danish apparatus as described in EPA recommended procedures (*5*). Both soil and water sample extracts were cleaned on a silica gel column, to remove interfering pesticides, using the method of Snyder and Reinert (*6*) followed by a sulfuric acid cleanup as described by Murphy (*7*).

All analyses were conducted by electron capture gas chromatography using a Varian 2760 instrument. A 1.8-m, 3-mm i.d., glass column packed with 1.5/1.95% OV-17/QF-1 on a chrom W-HP, 80/100 mesh support at 200 °C was used for PCB analysis. A 3% SP-2401 glass column of length 1.1 m and i.d. 3 mm was operated at 220 °C for analysis of PCT's. Mass spectrographic confirmation was obtained for selected samples. Quantitation of the PCB and PCT present was conducted by electronic integration of the area of all peaks matching the most closely related Aroclor standard.

Results and Discussion

PCB's were detected in soil samples from all three locations surveyed. The gas chromatographic elution patterns most closely matched those of Aroclor 1242 and Aroclor 1260. Decachlorobiphenyl was present in samples taken at two of the three sites. PCT's were detected in soils at the urban Chicago location and in sediments at the suburban Detroit location.

Manufacturing Facility. Aroclors have been produced by Monsanto Co. at the Sauget, Ill., site since prior to 1957. PCB mixtures ranging from 16 to 68% chlorine have been produced over the years. Production of the higher percent chlorine mixtures has been voluntarily reduced since 1970, and lower substituted isomers have been introduced. Polychlorinated terphenyls and chlorinated benzenes have also been produced at this facility. Terphenyl production was suspended in 1971 (*8*). The company operates a liquid injection incinerator to dispose of in-house liquid wastes and contaminated PCB's. The environs of the facility are dedicated predominately to heavy industrial use with a limited amount of residential housing.

The results of the analysis of soil samples taken in the vicinity of this manufacturing facility are summarized in Table I. Aroclor 1242 was present in approximately one-half of the samples. A maximum of 10 ppm Aroclor 1242 was detected 600 m to the northwest. Aroclor 1260 was present in all samples. Concentrations ranged from 0.03 to 9.6 ppm. Decachlorobiphenyl was also detected in all samples at a concentration ranging from 0.049 to 2.4 ppm. Total PCB concentrations of 0.15–20.7 ppm on a dry weight basis were detected within a

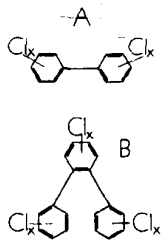


Figure 1. Basic chemical structure of (A) polychlorinated biphenyls (PCB's) and (B) polychlorinated terphenyls (PCT's)

Table I. Results of Analysis of Soil Samples Taken in Vicinity of Monsanto Co. Krumrich Production Facility in Sauget, Ill.

Direction from facility	Distance from facility, m	ppm Aroclor 1242	Aroclor 1260	Decachlorobiphenyl	Total PCB
SE	250	6.1	9.6	2.4	18.1
NE	450	0.68	0.31	0.38	1.37
SE	600	3.0	2.9	1.3	7.2
SW	600	<0.01	0.65	0.12	0.77
NW	600	10	9.6	1.1	20.7
NE	620	<0.01	1.4	0.90	2.3
NE	700	<0.01	0.12	0.61	0.73
NW	850	<0.01	0.03	0.40	0.43
NE	950	<0.01	0.05	0.097	0.13
SW	950	0.45	0.28	0.081	0.81
SE	1000	<0.01	0.20	0.27	0.47
SE	1050	0.82	1.3	1.6	3.72
NW	1100	0.46	0.39	0.12	0.97
SW	1250	<0.01	0.10	0.049	0.15
SW	1450	<0.01	0.26	0.081	0.34

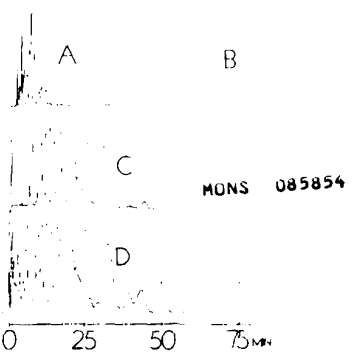


Figure 2. Typical chromatograms of standard Aroclors, decachlorobiphenyl, and soil sample taken in vicinity of Monsanto Co., Sauget, Ill.

(A) Aroclor 1242, (B) decachlorobiphenyl, (C) Aroclor 1260, (D) soil extract

1450-m radius of the facility. No PCT was present in soils from this location.

A typical chromatogram of a soil sample taken in the vicinity of the Monsanto facility is shown in Figure 2 along with the chromatogram of several other PCB's run under the same instrument conditions. This particular soil sample contains 11 ppm Aroclor 1242, 9.3 ppm Aroclor 1260, and 1.0 ppm decachlorobiphenyl. Lighter chlorinated Aroclor mixtures are likely present as well. The instrument conditions employed, however, do not give adequate separation of these mixtures to permit accurate quantification. The presence of decachlorobiphenyl at the concentrations observed is interesting. Decachlorobiphenyl has been reported as a component of only one Aroclor mixture (Aroclor 1268). Whether it may be a component of any of the terphenyl mixtures produced or a by-product is not known.

Referring again to Table I, the higher PCB concentrations are apparently at points closer to the facility with concentrations generally decreasing with increased distance. This distribution suggests airborne transport of the PCB's, including decachlorobiphenyl, from the manufacturing facility, with subsequent deposition onto the surrounding landscape.

A 24-h composite sample from the influent and the effluent of the Sauget village sewage treatment plant taken on February 17–18, 1975, was provided by plant personnel. The Monsanto Co. uses this facility jointly with the village. Analysis (μg/L) of these samples was as follows:

	Aroclor 1242	Aroclor 1260	Decachlorobiphenyl
Influent	6.8	8.7	<0.01
Effluent	4.5	4.7	<0.01

The treatment plant effects a reduction of 34% Aroclor 1242 and 46% Aroclor 1260. No decachlorobiphenyl was detected.

Investment Casting Facility, Chicago, Ill. Prior to 1972, Aroclors 5442, 5460, and 6090 were purchased and used by this industry in the compounding of investment casting waxes. Subsequent to 1972, Fenclor DK and Aroclor 5460 have been in use. Aroclors 5442 and 5460 are terphenyl mixtures with 42 and 60% chlorine by weight, respectively. Aroclor 6090 is 90% terphenyl and 10% Aroclor 1221. Fenclor DK is a foreign supplied PCB (Coffaro Corp., Milan, Italy) consisting of nearly pure decachlorobiphenyl. The manufacturing facility is situated in an urban neighborhood. Residential housing, parks, and a hospital are located within a 3-km radius.

The analytical results for soil samples at this site are tabulated in Table II. Aroclor 1260 was present in all samples, ranging from a low value of 0.22 ppm to a high of 1.8 ppm. Decachlorobiphenyl was present in all but two samples with a maximum concentration of 3.6 ppm located 20 m from the facility. Total PCB concentrations ranged from 0.22 to 5.2 ppm. PCT's were also present in many of these soil samples. The gas chromatographic elution pattern closely matched that of Aroclor 5460. One PCT concentration as high as 13 ppm was measured.

Figure 3A demonstrates that Fenclor DK is nearly pure decachlorobiphenyl. One minor peak elutes just before the predominate decachlorobiphenyl peak. The retention of the minor contaminant peak is such that it may be expected to be an octachlorobiphenyl since it exhibits the same retention time as the latest eluting peak in a standard Aroclor 1260 mixture. It is apparent by comparison of peak retention times with the sample chromatogram, Figure 3C, that decachlorobiphenyl is present in the soil sampled. The measured concentration of decachlorobiphenyl in this particular sample was 1.2 ppm. The presence of decachlorobiphenyl in this sample has been confirmed by mass spectrometry.

The sample elution pattern also indicates the presence of Aroclor 1260 as can be seen by comparison with the Aroclor 1260 elution pattern, Figure 3B. The concentration of Aroclor 1260 in this sample is 0.53 ppm. There is no indication of degradation of the Aroclor 1260 since virtually all peaks in the sample chromatogram are present in the same relative proportion as in the standard. There are no significant, undefined peaks in the chromatogram following the acid cleanup procedure. Certain of the sample chromatograms indicate the presence of lower weight PCB's; however, the quantities present tend to be far less than either Aroclor 1260 or decachlorobiphenyl at this site.

Figure 4 is a gas chromatogram of a soil extract from this site showing the presence of various PCB's, decachlorobiphenyl, and PCT. The PCT present closely resembles the Aroclor 5460 shown.

There exists no apparent spatial pattern of Aroclor 1260 distribution over the study site. There is little evidence in the data to indicate the source of the Aroclor 1260 contamination

Table II. Results of Analysis of Soil Samples Taken in Vicinity of an Investment Casting Facility in Urban Chicago, Ill.

Direction from facility	Distance from facility, m	ppm			
		Aroclor 1260	Decachlorobiphenyl	Total PCB	Aroclor 5460
NE	20	0.40	1.2	1.6	3.2
SE	20	0.26	0.51	0.77	0.38
SW	20	1.5	0.75	2.25	4.2
NW	20	1.6	3.6	5.2	13
E	450	0.69	0.58	1.27	4.1
NE	600	0.34	0.19	0.53	0.25
SW	620	0.57	0.040	0.61	<0.17
NW	620	0.29	0.001	0.29	0.28
NE	1050	0.81	0.033	0.84	0.17
SW	1150	0.67	0.020	0.69	0.79
NW	1220	0.56	0.034	0.59	<0.17
NE	1500	0.24	0.053	0.29	<0.17
E	1620	0.22	<0.001	0.22	<0.17
NW	1900	1.8	<0.001	1.8	0.23

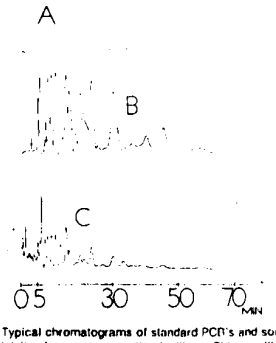


Figure 3. Typical chromatograms of standard PCB's and soil sample taken in vicinity of investment casting facility in Chicago, Ill. (A) Fenclor DK, (B) Aroclor 1260, (C) soil extract

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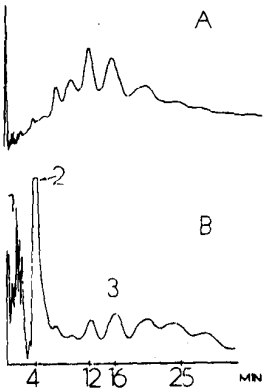


Figure 4. Chromatogram of Aroclor 5460 and soil sample taken in vicinity of investment casting facility in Chicago, Ill.
(A) Aroclor 5460, (B) soil extract showing presence of (1) PCB's, (2) decachlorobiphenyl, and (3) PCT's

Table III. Results of Analysis of Soil Samples Taken in Vicinity of Investment Casting Facility in Suburban Detroit, Mich.

Direction from facility	Distance from facility, m	ppm Aroclor 1260	ppm Aroclor 5460
SE	60	0.04	<0.17
NW	60	0.12	<0.17
NW	60	0.07	0.64
NW	200	<0.01	<0.17
SE	520	0.05	<0.17
NW	800	<0.01	<0.17
NE	900	0.14	<0.17
SE	900	0.06	<0.17
NW	1000	0.12	<0.17
SW	1050	0.03	<0.17
SW	1050	<0.01	<0.17
NW	1350	0.03	<0.17
SE	1500	0.04	<0.17
E	1650	18[a]	<0.17
NE	2100	<0.01	<0.17
SW	2100	0.02	<0.17

[a] Aroclor 1242.

is the facility surveyed. Aroclor 1260 has not, in fact, been reported to have been used by this company.

The spatial distribution of both decachlorobiphenyl and PCT do indicate higher soil-borne concentrations near the investment casting facility with generally decreasing levels at increased distances. There exists a strong statistical correlation among the data for decachlorobiphenyl and the PCT (Aroclor 5460) and little correlation between Aroclor 1260 and decachlorobiphenyl or the PCT.

Again, as at the production facility, the spatial distribution of the PCB and the PCT present is indicative of airborne transport. The Aroclor 1260 detected is apparently not from the source surveyed, whereas decachlorobiphenyl and PCT are likely from this user. It is somewhat surprising to see evidence of significant airborne transport of the higher substituted and less volatile PCB isomers and of PCT. That the gas chromatographic elution pattern shows little evidence of either selective emission or selective transport of the more volatile isomers would indicate the source of the airborne contamination is at such a temperature that the entire PCB or PCT mixture is in the vapor state at the point of emission.

Investment Casting Facility, Troy, Mich. This facility is located in a small industrial park in suburban Detroit, Mich. Surroundings consist principally of other small industries and residential areas. It has been reported that this company purchases the wax material used in the casting process from the company located in Chicago which is discussed above. The analytical results of soil samples taken at this location are summarized in Table III.

Aroclor 1260 is detectable in most of the soil samples. Decachlorobiphenyl is absent in the soils. PCT's are present in one sample taken 60 m northwest of the user. No spatial distribution pattern of PCB or PCT is evident. The one observed concentration of 18 ppm Aroclor 1242 appears to be anomalously high. Replicate analysis, however, yielded similar values for this sample. Aroclor 1242 was not detected in any other soil samples from this area.

A drainage ditch passes adjacent to the north boundary of the industrial park in which the facility is located. The drainage ditch serves to remove stormwater runoff in the vicinity and receives discharged cooling water from several local small industries including the one surveyed.

Analyses of cooling water at the point of discharge, of water and sediments within the drainage ditch, and of water and sludge deposits within the sanitary sewer system serving the area are reported in Table IV. It is apparent from these data that discharged cooling waters and waters in the drainage ditch at the time of sampling were free of both PCB's and PCT's. Bottom sediments within the drainage ditch, however, contained significant concentrations of Aroclor 1242, Aroclor 1260, decachlorobiphenyl, and Aroclor 5460. The source of the contamination of ditch sediments may be attributed to

Table IV. Results of Analysis of Various Samples Taken in Vicinity of Investment Casting Facility in Suburban Detroit, Mich.

Sample	Aroclor 1242	Aroclor 1260	Decachlorobiphenyl	Aroclor 5460
Cooling water discharge	<0.1 µg/l.	<0.1 µg/l.	<0.01 µg/l.	<0.1 µg/l.
Drainage water	<0.1 µg/l.	<0.1 µg/l.	<0.01 µg/l.	<0.1 µg/l.
Drainage water	<0.1 µg/l.	<0.1 µg/l.	<0.01 µg/l.	<0.1 µg/l.
Ditch sediment	2.3 ppm	6.7 ppm	0.09 ppm	6.7 ppm
Ditch sediment	9.4 ppm	8.9 ppm	0.11 ppm	5.1 ppm
Sanitary sewer water 1530, 2/22/75	<0.1 µg/l.	<0.1 µg/l.	<0.01 µg/l.	<0.1 µg/l.
Sanitary sewer water 1815, 2/22/75	<0.1 µg/l.	<0.1 µg/l.	<0.01 µg/l.	<0.1 µg/l.
Sanitary sewer water 1145, 2/23/75	<0.1 µg/l.	7.0 µg/l.	4.1 µg/l.	7.5 µg/l.
Sanitary sewer sludge deposits	<0.01 ppm	0.11 ppm	0.034 ppm	5.0 ppm

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wastewater discharges, sometime previous to the time of sampling, or to surface runoff in the vicinity.

Water and bottom sludge samples were taken from the sanitary sewer system serving the industrial park. Grab samples of the water were taken three times from a manhole access. As shown in Table IV, PCB's and PCT's were not detected in waters sampled at 1530 and 1615, but when sampled at 1145 the following day, PCB's and PCT's were present at a concentration of 11.5 and 7.5 μg/L, respectively. Sludge deposits within the sewer contained 0.14 ppm PCB and 5.0 ppm PCT on a dry weight basis. It appears that intermittent discharge of both PCB and PCT to the sanitary sewer system is occurring.

Conclusions

PCB contamination of the environment has occurred near the domestic production facility and two user facilities. The spatial distribution of soil contamination suggests airborne transport of the contaminant from the manufacturer and one of the two users surveyed. At the manufacturing site, Aroclor 1260 and decachlorobiphenyl were distributed in such a manner as to indicate the production facility to be the source. In urban Chicago, decachlorobiphenyl and PCT contamination appears to be superimposed on a rather random background of Aroclor 1260 contamination in such a manner to suggest the investment casting facility is the source of the decachlorobiphenyl and the PCT, but not the Aroclor 1260. In suburban Detroit, the background soil level of Aroclor 1260 shows no distribution pattern that would indicate the source to be the facility surveyed.

Influent and effluent wastewater samples from Sauget, Ill., attest to the removal, albeit incomplete, of PCB in the biological waste treatment system. Water and sediment samples from the suburban Detroit location indicate PCB and PCT discharges or contaminated runoff into surface drainageways sometime in the past. Discharges to the sanitary sewer system are apparently continuing.

Voluntary control on the release of PCB's and PCT's to the environment by industrial sources cannot be considered entirely effective. It is not possible to determine from soil contamination levels at the manufacturing site, whether or not the contamination has occurred since voluntary controls have been instituted (ca. 1970). At the urban Chicago location, however, it appears soil contamination has occurred since 1972 when Fenclor DK came into use. Contamination of wastewaters is still evident.

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Use of Permeation Tubes for Calibration of Vinyl Chloride Analyses

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■ A study was conducted to determine the performance of vinyl chloride permeation tubes when used to prepare low-level gaseous calibration standards which are convenient for the analysis of samples in the gas or liquid phase. Permeation rates were determined gravimetrically at three temperatures including extended periods at 25 °C. The rates at 25 °C for FEP and TFE Teflon tubes (0.250 in. o.d. × 0.030 in. wall thickness) were, respectively, 0.259 and 1.98 μg/cm min, and the activation energies of permeation were determined to be 15.0 and 11.9 kcal/g mol. Gas samples prepared in a flow dilution system containing a permeation tube were used to calibrate a gas chromatograph equipped with a gas sampling valve. Calibration data, accuracy, and limit of detection are presented. Although the permeation tubes were convenient, periodic weighing is recommended for highest accuracy since slightly decreasing rates were observed in four tubes and some polymerization of the reservoir liquid occurred in one tube.

Recent recognition of the carcinogenic hazards associated with vinyl chloride exposure generated the need to monitor low levels in work areas and ambient air. The federal Occupational Safety and Health Administration set an occupational exposure limit of 1 ppm for an 8-h time-weighted average with a ceiling of 5 ppm for a period not exceeding 15 min (*1*). An "action level" of 0.5 ppm and above was established for employers for implementing certain required control procedures. Because of important economic and legal implications, accurate analysis of airborne vinyl chloride is essential.

The Environmental Protection Agency recently published a tentative method of analysis for ambient air concentrations (*2*), similar to that recommended by the National Institute for Occupational Safety and Health for occupational areas (*3*). Both methods were based on adsorption of vinyl chloride on charcoal and desorption into carbon disulfide followed by gas chromatographic determination using a flame ionization detector. The accuracy of the method was thus dependent upon

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