

<Original> **Studies on the Preparation of Organic
Halogen Compounds Labelled by ^{38}Cl . (II)**

**A Thinlayer Chromatographical Separation of the Main
Component of the Organic Phase in Szilard Chalmer Reactions
of Aromatic Chloro Derivatives**

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Abstract

In a continuation of the 1st series of these studies, a separation of the components in organic phases obtained from Szilard Chalmer reactions of aromatic chloro derivatives was attempted to clarify the conditions of isolating the main component. Though it was unable to isolate all constituents of organic phases, the main component was well isolated by means of a simple distillation or a thin layer chromatography. It was identified that the main component was a recombined product of the recoiled ^{38}Cl atom with the original sample. In cases of solid samples the main component amounted about 80-60% of the radio activity of the organic phase, whereas the main component from liquid samples gave less than 70% of the radio activity of the organic phase. A prolonged irradiation could increase the yield of a main component, but it was, in case of a radiation sensitive compound, decreased as the irradiation is extended.

The number of side products separated from an organic phase of a liquid sample was greater than that of a solid sample, and hence the main component of a solid sample was isolated easier.

The overall yields of the labelled main products of 22 kinds of compounds were listed and a procedure of the separation of the main component by a thin layer chromatography was proposed and evaluated. The feasibility of the present procedure for labelling purpose was also reviewed with respects to the chemical structure of a compound.

요 약

본 연구의 제1보에 뒤이어서 방향족 염소유도체의 Szilard Chalmer 반응으로 얻어진 유기층의 성분을 분리하여서 표지 조건을 검토하였다. 여러가지 성분을 모두 분리

할 수는 없었으나 주성분을 증류법 및 박층 Chromatography 등으로 분리하였으며 그 화학구조가 원시료의 표지물임을 확인하였다. 고체시료의 경우에는 주성분이 유기표지물의 80-60%이었고 액체시료에 있어서는 70% 이하였다. 장시간 조사하면 주성분 수율이 증가 되었으나 방사선에 불안정한 화합물의 경우에는 도리어 감소하였다. Chromatography 로 분리되는 부생성물들의 수율은 액체시료의 경우 많았으나 고체의 경우에는 이 보다 적었고 따라서 주생성물의 분리가 용이하였다. 각 화합물의 표지 수율을 표시하고 Chromatography 에 의한 분리조건을 논의하였으며 이 표지 방식의 실용성을 화학구조와 관련시켜 고찰하였다.

1. Introduction

In a continuation of the previous series of these studies¹⁾, the organic phase from the irradiated compound was examined for components of an organic structure. Generally, the number of components of an organic phase from a neutron irradiated organic compound had been expected to be numerous due to a complicate radiolytic scission of chemical bonds involved in a molecule, and consequently the separation of the organic fraction of the reaction had been investigated by means of a radio gas chromatographical technique^{2, 3)}. A complete identification of each fraction of an organic phase had nevertheless been attainable by combining very complicate sets of eluating reagents in a column³⁾.

Since the scope of this study is to prepare labelled compounds themselves with a relatively simple and convenient procedure of isolating the desired component of the irradiated target materials, the main effort may better be concentrated to separate the principal component of the highest radio activity from the organic phase. Furthermore, the separated component should easily be collected to be used for a practical application. Considering these aspects of the study, a general technique of isolating a trace quantity of organic compound was expected worthwhile to be investigated. The conventional method of a simple distillation with a radioactive carrier may be useful for the case of a volatile

component of a relatively low boiling point, and a radio thinlayer chromatographical method would be effective and convenient for isolating and collecting the trace quantity of a labelled component for the case of a non volatile component of a high boiling point.

Isolations of the main component of a neutron irradiated organic material were reported by means of the preparation of a derivative with the functional group in the molecule⁴⁾, by means of a thinlayer chromatography⁵⁾, by means of a paper chromatography⁶⁾, and numerous cases of others were reported in the literatures. However, the isolation of the main component from the irradiated aromatic chloro derivatives had not yet been studied in details, and hence a considerable effort may be necessary for the purpose proposed. A careful selection of a solvent system of a developing solvent and an adoption of a proper coating and supporting media of a chromatographical plate would be essential to attain a well resolved peak of a main product on the chromatogram. And an efficient and rapid way of identifying the chemical structure of each fraction of a distillation process would be necessary for the purpose, since the half life of ^{38}Cl is only 37 minutes. The duration required for the process of the isolation procedure should also be minimized as long as possible to save the radioactivity in a molecule.

In the 1st series of these studies, the general

tendency of a recoiled reaction of the present case had already been investigated relating the chemical structure of the compound, and components of the organic phase were estimated to be not complicate in both cases of a solid sample and a liquid sample irradiated for a longer period. It may, therefore, be interesting to correlate the main component separated by the proposed method to these trends, though a minor component may sometimes not be identifiable. In this paper a thin layer chromatographical study of the organic phase was reported and the feasibility of the method for a practical application was reviewed. A radiolytic insight of the recoil reaction of ^{36}Cl was also attempted, wherever it may be possible.

2. Experimental

(1) Materials

The reagent grade of compounds used in this experimental were obtained from a commercial source (TOKYO KASEI). All compounds were checked their melting points, refractive indices, chromatographical data, and spectrophotometric identifications, and they were purified by a distillation or a

recrystallization wherever an extended purification was required for an irradiation experiment.

(2) Extraction of the inorganic portion and separation of the organic phase thereof

The procedures described in the previous series of this paper¹⁾ were followed and the last portion of the organic phase of the process was examined for the components as follows.

(3) Separation of the main component of an organic phase

(a) A distillation process

The organic phase separated as described in paragraph 2 of this experimental was taken in a small distillation flask and 5-10cc of the sample compound itself was added to the flask as a carrier. The resulting content of the flask was distilled under the ordinary pressure and the each fraction was evaluated for its activity and chemical identity as follows:

Example:

Sample: Monochlorobenzene

Irradiation time: 1 minute (30 minutes annealing)

Counting time: 1 minute.

Fraction	Volume ml.	b. pt. °C	Radio Activity per ml. c. p. m.	Total Activity c. p. m.	Refractive Index* n_D^{20}
1	15	~80	6642	99,630	(1.5012)
2	10	~80	12402	124,020	(1.5037)
3	8.7	80~	83809	729,138	(1.5207)
4	15	Ether wasing of distilling residue.	4768	71,520	—
5	15	Ether washing of cooler.	8418	51,270	—
6	15	Water washing of distilling residue.	7143	10,145	—

$$\% \text{ of main component}^{**} = \frac{\text{Total activity of (3)} \times 100}{\text{Total activity of (1+2+3+4+5+6)}} = 66\%$$

*Uncalibrated

Benzene (Authentic) = n_D^{20} 1.5012

Monochloro benzene (Authentic) = n_D^{20} 1.5212

**The yield of main component would better be determined by a gas chromatographical method, but the separation of the main component labelled for practical uses would be more convenient by a simple distillation.

(b) Radio thinlayer chromatography

(i) Developing and identification of the main component:

The organic portion separated by this experimental (see paragraph 2) was dried with anhydrous calcium chloride or magnesium sulfate and filtered. The definite amount of the dried sample was spotted in a thinlayer plate and developed in an ascending way with a proper solvent system in a mess cylinder of 500-250 cc. with a tapered cock. The plate was dried at a room temperature when it was developed more than 10 cm from the spotting line. The plate was then cut in a 0,5cm piece from the spotting line, and each portion was taken in a test tube. Each test tube was counted for its radio activity by means of a γ -Well Type Scintillation Counter (Fuzitsu, TM-II) and R_f value of the main component of the sample was determined from the evaluation of the radio activities obtained. Separately, the authentic sample was checked for its R_f value under the same solvent system and was compared with the former. The chromatographical data of each sample were given in the Table 2.

(ii) The determination of labelling yield of the main component: On basis of radio activities obtained from the above, a radio thinlayer chromatogram was plotted and the radio activity of sections composed of the peak of the main component on the chromatogram was compared with the total radio activities on the plate. An example was shown in the Fig. 1-2.

Labelling Yield of Main Component(%) =

Total Radioactivity of Sections Composed of the Main Peak $\times 100$ / Total Activity on the Plate.

In Table 1 the labelling yield for each compound was given together with the organic yield.

(4) Determination of the radioactivity of the main component labelled:

The counting efficiency of the γ -well type scintillation counter for the radio activity of ^{38}Cl was determined by means of an authentic sample of $\text{NH}_4^{38}\text{Cl}$. Based on the data of the labelling yield of the main component and total radio activity of the organic portion separated from the irradiated target, the approximate amount of the radioactivity of the main component labelled was calculated as follows.














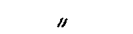

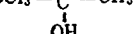



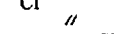

Radioactivity of main component(c. p. m.) = Total radioactivity of the organic portion \times labelling yield of the main component / Counting efficiency.

The calculated value was corrected for the radioactive decay occurred during the course of the processing the sample and was again converted to an unit of curies per gram. In Table 3, the corrected value of the radio activity of the main component of a sample at the end of the reactor irradiation was partly given.

3. Results and Discussion

Results obtained from the separation of the organic phase from each irradiated aromatic chloro derivative are summarized in the Table 1. The organic yield multiplied by the percentages of the main product was listed in the table as the overall yield of that component. As stated in the previous paragraph, the separation of aromatic chloro derivatives irradiated in the reactor had not yet been investigated for the particular type of the chemical structure treated in this study, and hence the method of separating the main component was, in particular, the important aspect of the present investigation. In the following, various trends of the separation procedure are, at first, discussed with respects to the functional group of the com-

Table 1. Yields of recombined products of the recoiled ^{36}Cl atoms in neutron irradiated organic halogen Compounds

Type of Compounds	State of Sample	Irradiation Time (min)	Organic Yield (%)	Yield of Recombined Products(%)	Overall Yield Labelled(%)	Remarks
	Liquid	30	62	70	43.4	
"	"	15	62	71	44.0	
"	"	1	45	70	31.5	
	"	10	89	66	58.7	
"	"	1	75	61	45.8	
	Solid	1	90	60	54.0	
"	"	10	89	62	55.2	
	Liquid	10	86	65	55.9	
	"	10	91	51	46.4	
	Solid	10	93	52	48.4	
	"	10	90	53	47.7	
	"	10	93	62	57.7	
"	"	1	92	73	67.2	
	Liquid	15	70	73	51.1	
	Solid	10	36	74	26.6	
	"	10	31	49	15.2	
	Liquid	10	60	61	36.6	
	"	10	54	71	38.3	
	Solid	10	46	87	40.0	
	"	10	84	65	54.6	
"	"	1	88	40	35.2	
	Liquid	15	64	50	32.0	
	Solid	10	46	65	29.9	
"	"	1	35	69	24.2	
	Solid	10	39	65	25.4	
"	"	1	38	46	17.5	
	"	10	47	52	24.4	
	"	10	52	74	38.5	
"	"	1	53	59	31.3	
	"	10	55	66	36.3	
"	"	1	42	43	18.1	

















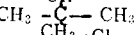




pond, since the separation by either a simple distillation or thinlayer chromatography have more or less been related to the functional

group in a molecule.

(1) Phenyl Chlorides.

As described in the section (a) of the

Table 2. Radio thinlayer chromatographic data of the main products from irradiated organic chlorine copounds

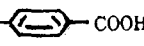
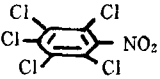
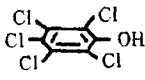
Type of Compounds Irradiated	Radio Thin layer chromatographic data			Remarks
	Type of Plate	Developing Solvent (v/v)	R _f Value of the product	
	S*	Hexane: Acetone (2:1)	0.53	Peak at R _f =0.39
	A*	Hexane: 2-Picoline (1:1)	0.63	
	A	"	0.44	
	A	Hexane: Pyridine: picoline	0.65	
	A	Hexane+Picoline (1:1 v/v)	0.69	
	A	Hexane+Pyridine (1:1)	0.63	
	A	"	0.56	
	A	Hexane:Pyridine:Picoline(2:1:1)	0.13	
	A	Hexane+Pyridine+Picoline(2:1:1)	0.25	
	S	Benzene+MeOH (9:1)	0.25	
	S	Hexane+Acetone (2:1)	0.53	
	S	"	0.53	
	S	"	0.47	
	S	Benzene+MeOH (1:1)	0.37	
	S	Hexane+Acetone (2:1)	0.63	
	S	Hexane+Acetone (2:1)	0.43	
	A	Hexane+Pyridine (1:1)	0.11	
	S	Hexane+Acetone (2:1)	0.47	
	S	Hexane+Acetone (2:1)	0.50	
	S	Hexane+Acetone (2:1 v/v)	0.44	
	S	Benzene+MeOH (9:1v/v)	0.60	

* S represents a Silica gel coated, Merck Kieselgel, and A represents alumina coated, Merck F 254, Neutral (Type T), respectively.

paragraph 2 of the experimental, a simple distillation of the organic phase from the irradiated monochlorobenzene could give a good separation of the labelled monochlorobenzene radiochemically. The percentage of the fraction amounted 66%. There were some portions of the distillation residue in the flask, which may be composed of non volatile fractions of the organic phase such as polychlorobenzene and some biphenyl chloro derivatives as reported in previous papers¹⁾. The isolation of the main component may, of

course require, further strict identification of the contents of the main fraction of the distillation by means of instrumental methodes, but the refractive index showed a slightly discrepancy at the fourth place below decimal point when it is compared to the standard sample. It was, therefore, considered pure enough to be used for an application, since the other volatile products from the recoiled reation of this compounds are of aliphatic structure resulted from the radiolytic decompositions of the benzene ring, the refractive

Table 3. Yields of organic and recombined products of the recoiled ^{35}Cl atoms in a neutron irradiated organic halogen compound for tracer applications

Type of Compounds	State of Sample	Irradiation Time (min)	Organic Yield (%)	Yield of Recombined Product* (%)	Overall Labelled Yield (%)	Remarks
Cl-  -COOH	Solid	5	39	65*	25.4	
//	//	10	36	67*	24.1	
//	//	15	52	52*	27.0	
//	//	20	46	59*	24.1	
//	//	30	41	55*	25.3	14.420 mci/g
Cl-  -NO ₂	Solid	5	70	—	—	
//	//	10	72	83***	59.8	
//	//	15	70	83***	58.1	
//	//	30	73	89***	65.0	49.980 mci/g
//	//	90	70	63	44.1	
Cl-  -OH	//	1	49	—	—	
//	//	10	56	66**	37.0	
//	//	15	59	52**	30.7	
//	//	20	57	70**	39.9	
//	//	30	60	49**	29.4	27.470 mci/g
Tetra-chloro fluorescein	//	15	—	—	20.0****	
	"	30	28	—	21.0****	9.430 mci/g
5-chloro-Uracil	//	1	—	—	17.0*****	
Uracil	//	10	—	—	21.0*****	
Uracil	//	30	14	59	8.3*****	

* The main product was separated by the following ways of a thinlayer chromatography.

* Plate: Silica Gel coated. (Merck F-254)
Solvent: Butanol, HOAc, and H₂O (4:1:1 v/v)
R_f: 0.65

** See Fig. 2

*** Plate: Silica Gel coated
Solvent: Hexane : acetone (2:1 v/v)
R_f: 0.55

**** Plate: Silica Gel coated
Solvent: Butanol saturated by 5% HOAc sol.
R_f: 0.73

***** Plate: Silica Gel coated
Solvent: Butanol saturated by 5% HOAc sol.
R_f: 0.63

indices of which are usually below than 1.5000⁷⁾. According to observations based on

refractive indices of fractions obtained from the distillation, it revealed further that the

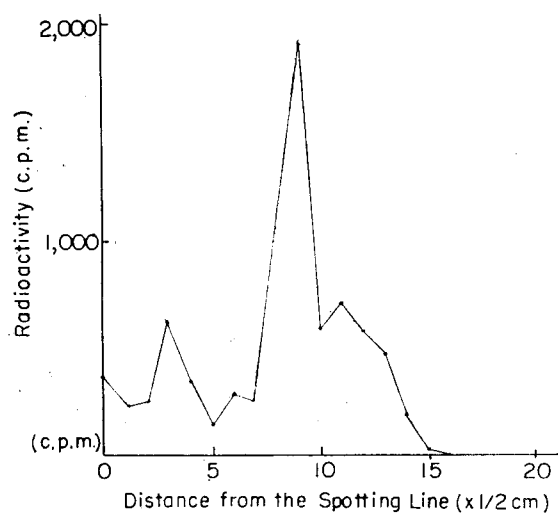


Fig. 1. Thinlayer chromatogram of the organic peak from the neutron irradiated monochlorobenzene.

Plate: Al_2O_3 coated, Merck F-254 Neutral (Type-T)

Solvent: Hexane : 2-picoline (1:1 v/v)

Temp.: 24–26°C

organic phase composed of 20% of a volatile product originated from the decomposition of a molecule, 66% of monochlorobenzene, and 12% of poly or dimerized chloro aromatics.

As shown in the Table 2, the separation of the organic phase revealed that a particular solvent system for a particular structure of phenyl chlorides was required for an efficient separation of the desired main product. Various systems of a developing solvent and both silica gel and alumina coated plates were tested for the separation of the components of each sample, but the solvent systems and plates listed in the table 2 were proved to be the best approach for the purpose. Since the electronic moieties of benzene rings of these compounds differ slightly from each other, the structural sensitivities of mono-, di-, tri-, and tetra- chlorobenzenes towards a chromatographical media or solvent system are seemed to be not distinct enough to separate all series of these compounds in a developing solvent and a plate.

An activated alumina grazed in a fine powder was known to form a complex with a π electron donor under a proper solvent system⁸⁾, and consequently the separation of aromatic derivative using this medium of a thinlayer plate is expected to be effective enough to overcome the structural resemblance of compounds. However, the selection of the proper solvent system was extremely of time spending efforts. The present results were, therefore, obtained after a tedious work of selecting a solvent system. Probably, basicity differences between pyridine and picolidine in the solvent system adopted could coupled with the slight differences of the π electron density of the benzene rings of these series of the compounds to afford a separation of the component. In the Fig. 1, an example of the thinlayer chromatogram was given which may be able to explain the delicacy of the separation mode of the system. As shown in the figure, the main products are slightly separated from the secondary side product at a narrow range of R_f value. However, it could show that the other radiolytic or radiation reaction's products are of minor in respects to their radio activity, and the determined yield of the main component was resembled to that of a simple distillation. In cases of di-, tri-, and tetrachlorobenzenes, the same pattern of the chromatogram as that of monochlorobenzene could be obtained, though the solvent system should strictly be selected for an individual compound. On the nature of the peak obtained on the chromatogram, some detailed informations may be required to assure the presence of a single compound in the peak. For this purpose, the R_f value of the product was compared to that of an authentic sample to give a well coincidence between two R_f values. In the point of elucidating the feature of the recoiled

reaction of the system accurately, a radio gas chromatographical technique would be preferred provided that an efficient charging reagents of the column is readily available, since the great differences of the physical constants of the present compounds could considerably contribute to a fine resolution of the component in addition to the structural differences. However, it may require a complicate set of charging materials, a rapid scanning and temperature programming systems for a time consuming elution of the column which retains a component of a very high boiling aromatic component, and a collecting system of each fraction with a large set of a preparative column. It may, therefore, be said that the present procedure of the separation is of very convenient and simple, which could easily be applicable for labelling purposes under the usual circumstances of a scientific laboratory.

2) Chloronitrobenzenes:

As partly shown in the Table 2, the separation of the components in this series of compounds were easier than those of chlorobenzenes since the electron density on the ring is well apart by the chemical structure of the deactivated benzene ring with nitro substituents. A reported solvent system⁹⁾ could well be applied using a silicagel coated plate. However, a slight modification in the proportion of a solvent in a system was required for chloronitroanisole derivatives as shown in the table 2. Generally, the chromatogram showed very minor fractions of side products, and R_f value of the main peak coincide with that of an authentic sample.

(3) Chloroanilines and Chlorotoluenes

Both chloroanilines and chlorotoluenes with an activated benzene ring could separate by means of the solvent systems reported for the separation of anilines and nitrochloro-

benzenes¹⁰⁾. This may be due to the distinct electron moieties of the benzene ring of these compounds. Again, the chromatographical pattern of the separation showed a distinct peak of the main product with a minor peaks of the side product, and the R_f value of the main peak was well coincided with that of an authentic sample.

(4) Benzylchlorides and Other Aliphatic Chlorides

These compounds were separated by the solvent systems adopted in section 3, 2, and 1 of this discussion. In case of benzyl chloride it was expected that some ring chlorinated species of products may be involved in the organic phases due to a radiation chlorination of the benzene ring by the recoiled ³⁸Cl atom, and hence the organic phase was examined by the procedures of separating phenylchlorides to locate these products on the chromatogram. It was, however, observed that there were very minor radioactivity at the R_f regions of phenylchlorides on the plate. Probably, the chlorination of this kind could not be effected under the present condition of the irradiation. Conversely, chlorotoluenes were examined for the presence of the side chain chlorides which may be produced by the radiation chlorination of methyl group on the ring. Again, the side chain chlorides could not be detected in an appreciable radioactivity on the plate. Since the side chain chlorination of a toluene under a prolonged ionizing radiation is reported previously^{11, 12)}, the present method of the irradiation was not sufficient enough to effect the side chain chlorination of the ring with respects to the irradiation time. Generally, the chromatographical pattern of the separation on the plate showed a distinct main peak of the labelled benzylchloride and chlorotoluenes with a minor peaks of various side products.

(5) Some Tracer Compounds*

As shown in the Table 3, *p*-chlorobenzoic acid was separated by means of a solvent system reported for a paper partition chromatography⁶⁾. The separation of the main product was well achieved, but a considerable radioactivity was accumulated at lower R_f values on the plate than that of the main product. Probably, these may be composed of a complicate mixture of the decarboxylation product of the compound as proposed in the previous paper¹⁾. Pentachlorophenol was well separated by the reported procedure⁹⁾, but there was a significant radioactivity at the higher R_f region on the plate than that of the main peak, as shown in the Fig. 2. This may be the dimerized or polymerized product of the phenol, since phenol could be polymerized or dimerized under a severe

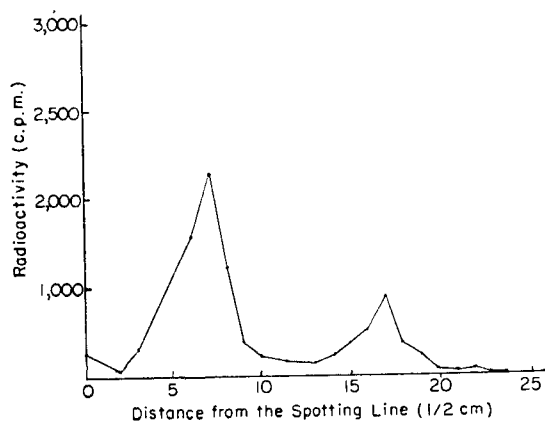


Fig. 2. Thinlayer chromatogram of the organic phase from the neutron irradiated pentachlorophenol.

Plate: Silica Gel, Merck Kieselgel(F-254)
Solvent: Hexane:acetone (2:1 v/v)
Temp. 22–24°C

* In these compounds they are soluble in an alkaline solution and therefore they were crystallized once from alcohol to obtain a crude matrix of the organic portion. The crude matrix was examined by the thinlayer chromatography. The organic yield listed in the table 3 was determined from the radioactivity of the crude matrix and the mother liquor on basis of the same method of the calculation described in the experimental.

radiation. The detailed identification of this peak was, however, expected in a future investigation. Tetrachlorofluorescein was again separated by the solvent system adopted for the case of *p*-chlorobenzoic acid. Uracil chloride(5-chloro derivative) was also separated by the same solvent, but a considerable radioactivity was accumulated at the lower R_f region on the plate than that of the main peak. Presumably, these may be composed of various decomposition products of the pyrimidine ring under the radiation.

(6) Yields of the Main Product

Generally, the yield of the main product of the irradiated aromatic chlorides showed an increasing trend at a prolonged irradiation time, but it was decreasing in unstable or polychloro derivatives as the irradiation is extended. Reasons for these observations were not clear for the time being, since this tendency was quite of random to be defined. It may, however, be said that polychloro derivatives or unstable compounds would produce more complicate fragments of molecules during the course of the irradiation which may produce many numbers of minor side products with recoiled ^{38}Cl atoms at the sacrifice of the main products, as shown on the chromatograms. Researches along these lines may be very interesting to be explored with respects of a radiolytic reaction of compounds.

In the Fig. 3 the general tendency of the changes of the yields of main products for some tracer compounds was plotted against the irradiation times. The plotted curve may be useful to predict the yield of a labelled product and may practically serve as a standard for checking the irradiation time suitable for obtaining the highest yield or a rapid labelling at the expense of the yield depending on the particular situation of the proposed labelling works.

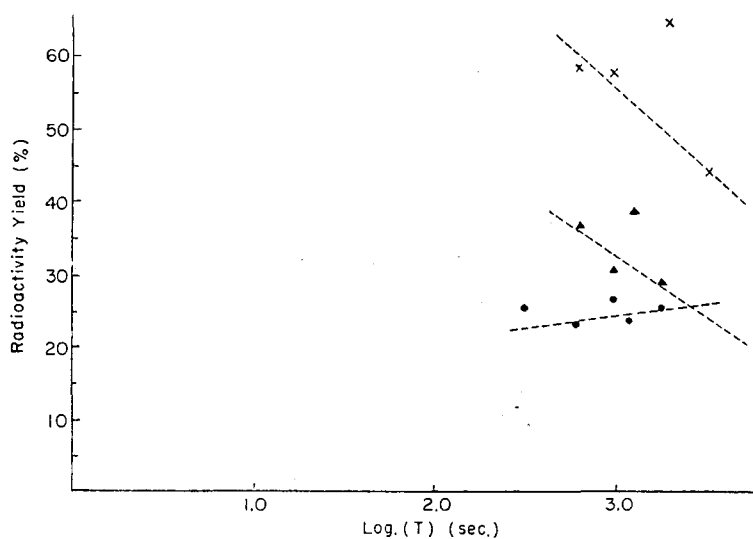


Fig. 3. The overall yield of the labelled product *v.s.* the irradiation time for some tracer compounds

Points on curves denote the following: ○ denotes p-chloro benzoic acid, × denotes pentachloronitro benzene, and ▲ denotes penta-chloro phenol, respectively.

(7) Specific Radioactivity of the Product.

The radioactivity of the main product calibrated for the radioactive decay and counter efficiency was listed in the table 3 as examples. Since the reaction is a $n-\gamma$ type of a nuclear reaction, the radioactivity of the labelled product may easily be increased as the amount of the sample is increased without any significant modifications of the organic yield or labelled yield. Author may propose to use a number of polyethylene tubes containing 0.2-0.4g. of the sample and irradiate them together in a rabbit tube, when a high gross radioactivity of the product is required for the application. On the specific activity of the product, the value listed in the table 3 may be the upper limit obtainable for a sample at a irradiation time. However, the processing time and developing procedure may be shortened to save the radioactivity, whenever the situation require to do them to increase the specific activity of the product.

* The total time for processing a sample in the present procedure was about 2hrs.

In conclusion a procedure of labelling organic chloro derivatives could be achieved by a simple distillation or thin layer chromatography of the organic phase of neutron irradiated compounds in the reactor. The general tendency of the labelling reaction, irradiation time, yield of the main product, and radio activity of the product could be predicted in advance to evaluate the feasibility of labelling works for an application.

Though the present study had been conducted without degasing the sample in a polyethylene tube and the yield or other parameters are not consistent enough to elucidate the reaction mode quantitatively, the observations and discussions obtained during these studies would be valuable to understand the general mode of the recoiled reaction of these types of compounds, which were not extensively explored.

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