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Study on a Ridio Paper Partition Chromatography of Organic Halogen Compounds by a Neutron Irradiation.

A Qualitative Approach

By

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有機하로겐 化合物의 中性子線 照射에 依한 定性放射化 크로마토그래피에 關한 硏究

原 子 力 硏 究 所

金 裕 善 · 崔 松 子

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要 約

展開한 페이퍼크로마토그램을 韓國 Triga-Mark-Ⅱ 硏究用 原子爐의 뉴마딕튜부施設(中性子線束; 1.5×10^{12}n/cm^2. sec)을 利用하여 照射하여본 結果 크로마토그램上에 展開된 스폿트(spot)를 定性的으로 確認할 수 있었다.

有機하로겐 化合物(크로로酸, 크로로에스테르, 沃化物, 及 弗化物)에 있어서 一般發色法으로서는 明確한 着色 스폿트(spot)를 나타내지 못한 것에 있어서도, 이 方法으로서 展開된 스폿트를 明確히 區別할 수 있었다.

濾紙成分의 放射化에 依한 誤差를 減少시키기 爲하여 濾紙두께 補正 及 放射化度 補正을 硏究한 結果, 濾紙두께 補正 及 放射能崩壞 補正法을 究明하였으며, 이 方法으로 스폿트를 放射化하여 定性分析하는데 좋은 結果를 얻을 수 있었다.

有機하로겐化合物의 分析 及 確認에 있어서의 이 方法의 效用性에 關하여 論議하였다.

濾紙相으로는 正常相 及 逆相을 함께 使用하였다.

Abstract

When a developed paper partition chromatogram was irradiated by means of the pneumatic tube system of the Korean research reactor (neutron flux: 1.5×10^{12}n/cm^{2}sec.) the qualitative confirmation of the developed spot on the chromatogram was possible. In the case of an organic halogen compounds (chloro-acid, chloro-ester, iodide, and fluoride) the spot analysis was possible by the present method whereas the same spot could not give the distinct coloring with a common coloring reagents. Filterpaper thickness calibration and activity calibration induced by irradiation of the components of the filter paper, which were a source of erraneous interpretation of the spot, were

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searched and an average filterpaper calibration method and filter paper activity were improvised to obtain a good qualitative analysis of the spot. Finally the use and applicability of this method for the analysis and identification of an organic halogen compound were evaluated. As the filter paper phase an ordinary phase (Whatmann #1, filter paper) and reversed phase (liquid paraffin impregnated) were used.

Introduction

Even though various methods of evaluating the spot developed on the paper partition chromatogram were searched, a radio chromatography of direct irradiation under an ionizing radiation source is not yet completely explored. An isotopic diluted sample [1] were used as the sample and the sample was developed on a filter paper, the activity of which was evaluated by means of the direct counting or chromatography scanning devices. [2] Few cases were reported for the radiochromatography by means of direct irradiating the developed chromatogram under a low level neutron irradiation source for the case of amino acid and chlorin containing phenyl derivatives. [3] When the radio chromatography was evaluated there were some difficulties to interpret the activity induced chromatographic peak because of the background of the activated filter and activity difference caused by the difference of the filter itself. [4] The present research was, therefore, concerned to develop an effective method of radio chromatography applying the high level neutron source which will be helpful to produce a considerable quantity of a short or medium life isotope on the developed spot of a paper chromatogram. Especially organic compounds, which was known to be inert to a coloring agent and hence difficult to be evaluated by usual spot analysis technique of coloring, were chosen as the sample. For instance halogen compounds, the coloring agent of which is usually silver nitrate or ferrous suplphate, will be very difficult to be evaluated by the ordinary paper chromatographic technique, because fluoride or polyhalogenated compounds is inert to produce a distinct color by silver nitrate reagent. The qualitative appoach for the spot analysis of the developed paper chromatogram of various organic halogen compounds was, therefore, undertaken and the result reported. The method will be helpful to understand the paper partition chromatogram of organic halogen compounds, which is difficult or not efficient to be analyzed by the conventional coloring technique.

Experimental

Starting materials. The sample used in the present research was mainly obtained from commercial source (Fisher certified and Wako Chemicals). Solvent was once distilled and the purity was checked prior to use. Filter paper was obtained from Whatmann #1 chemically prepared output. The reversed phase filter paper was made at this laboratory by means of developing the filter paper (without spotting sample) with a 5% solution of liquid paraffin in petroleum ether..

Paper partition chromatography

Ordinary phase: Whatman #1 filter paper chemically prepared was used. A sheet of filter paper

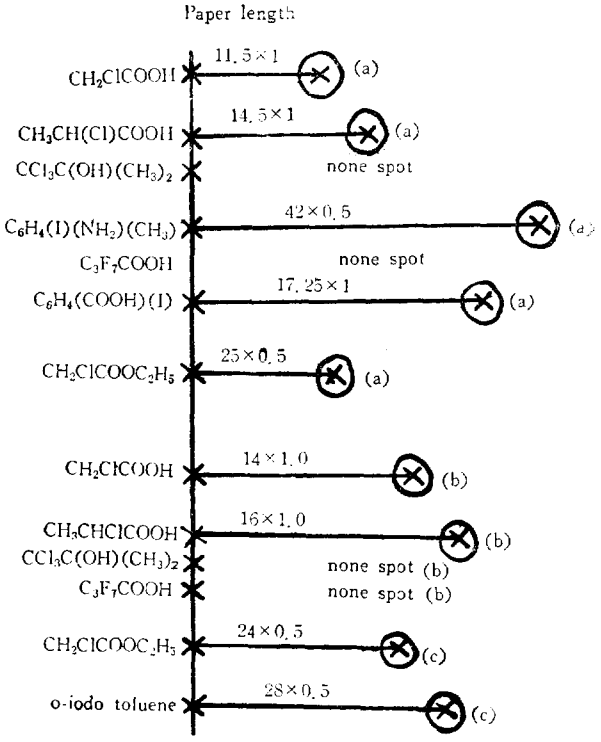


Fig 1, 2, 3. Paper chromatogram of organic halogen compounds.

(a) Phase, ordinary. solvent, 2-propanol: pyridine: water (8:1:1). Coloring agent silver nitrate solution in ammonia.
(b) Phase, ordinary. solvent, ethanol: pyridine: water (8:1:1); coloring agent, same as (a).
(c) Phase, Reversed; solvent; ethanol (aq.). Coloring agent, same as (a).

was taken and the filter paper was cut in the scale of 30 cm × 23 cm square at the corner of the sheet in the paper fiber direction. Sample was spotted at the interval of 2.5 cm excluding both marginal position. (See Fig 4) Total six spottings were made on a sheet. The spotted filterpaper was developed during the course of 6-8 hours in a chromatograph chamber of a Pyrex Glass Jar (height 35 cm dia. 15 cm) with a cover. The solvent used was a solution of ehtyl alcohol, pyridine and water (8:1:1), 2-propanol: pyridine: water (8:1:1:), ethyl alcohol, acetic acid, and water (8:1:1), and benzene, sodium carbonate, and butyl alcohol (saturated). The best chromatogram was obtained in the case of ethyl alcohol and propanol systems. The developed chromatogram was sprayed with an ammoniacal solution of 0.1 N silver nitrate. The spot analysis was made after exposing the chromatogram under sunlight for 5-10 minutes. The chromatogram of organic halides were given in Fig 2.

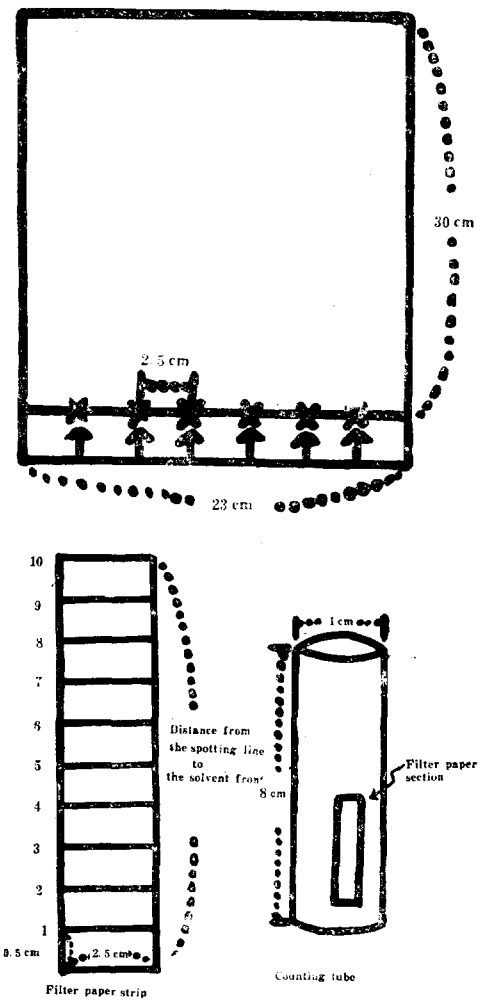


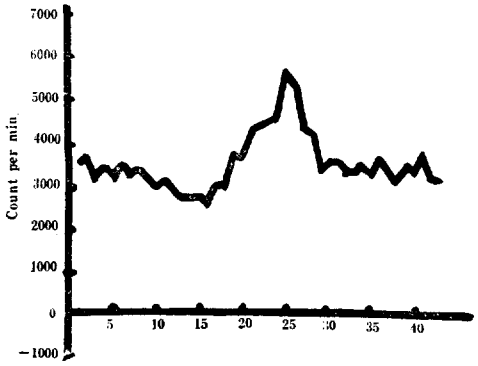
Fig 4. Filter paper

Reversed phase. A sheet of the filter paper described above was cut in the same size and same direction. The filterpaper was developed without spotting a sample with a solution of 5% liquid paraffin in petroleum ether. The processed filterpaper was then dried at room tempemature (20—25°C). The sample spotting was the same as the ordinary phase. The spotted filterpaper was developed with ethanol using the predescribed chromatographic chamber during the course of 8—12 hours at the room temperature. The developed filterpaper was dried in the oven at 80°C and sprayed with an ammoniacal solution of 0.1 N silver nitrate. The spot was identified after exposing the filterpaper under sunlight during the course of 5—10 minutes. This chromatogram was shown in the Fig 3.

Radiochromatography under neutron irradiation. The sample was spotted and developed as described in the above. The developed paper chromatogram was dried at 80°C for a few hours, and it was cut in the pieces of 2.5 cm width. Care was taken to centerize the sample spotting point in the middle of the paper. The distance from the spotting line to the solvent front was taken as the height of the irradiating sample filterpaper (see Fig 4). The filterpaper was sectioned in an equal width of 0.5 cm and the each section was folded and irradiated in a nylon rabbit tube under the neutron flux of 1.5×10^{12} n./cm^2, sec. for two to five minutes. The irradiated paper was cut in a pieces along the predefermined section and stored in a pyrex test tube (8 x 1 cm). The tube contained the filter paper strip was counted for one minute by means of a well type scintillation counter (Tracer Lab.)

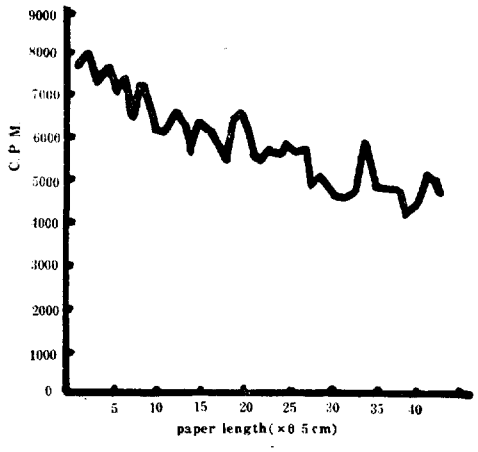
The activity induced on the each section of the paper strip was recorded together with the section number and the time interval from the irradiation time. From the data obtained from the activity counting various radiochromatogram were obtained according to the following methods.

No. 1. Short time irradiation (2minutes)
$CH_2ClCOO_2H_5$ (solvent: 2-propanol: pyridine: water (8:1:1)



Paper length (×0.5cm)

No. 3. Blank filter paper
(irradiated for five minutes).



A. Direct method.

The activity measured for each paper script was plotted against the paper length(see Fig 5). For the thickness calibration, the filter paper developed without spotting the sample was processed as the same manner and plotted against the paper length. The activity induced by the blank filter paper was substracted from the activity of the sample chromatogram and the difference in the activity was plotted against the paper length of the sample filter paper(see Fig 5 chromatog ram #1, #2 and #3).

No. 2. O-iodo-toluene (reversed phase).

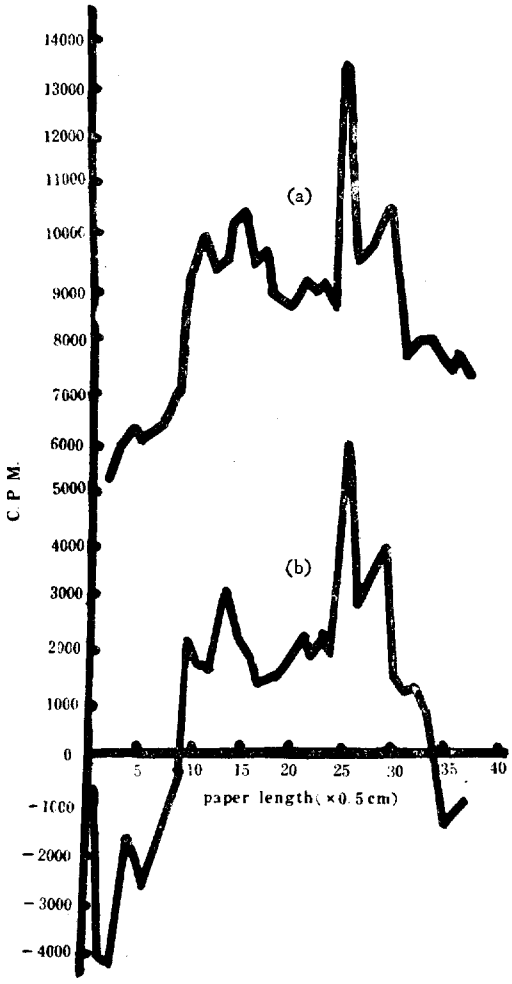


Fig 5. Direct and thickness calibrated radio chromatogram
(a) Direct radiochromatogram
(b) Filterpaper thickness calibrated

B. Alternate thickness calibration methods.

Two chromatogram were prepared. One of the two was the blank filter paper, which was developed by the same solvent as that of the sample chromatogram. The sample and blank chromatographic strips were irradiated simultaneously in one batch. The sample chromatogra phy and blank were counted alternatively, so that the activity of the sample paper and the blank of the same number of the section can be counted at the interval of one minute or so. The difference between the sample activity and the blank activity was plotted against the paper lenfth of the sample paper (see Fig 6).

The counting time of each section was 1 minute.

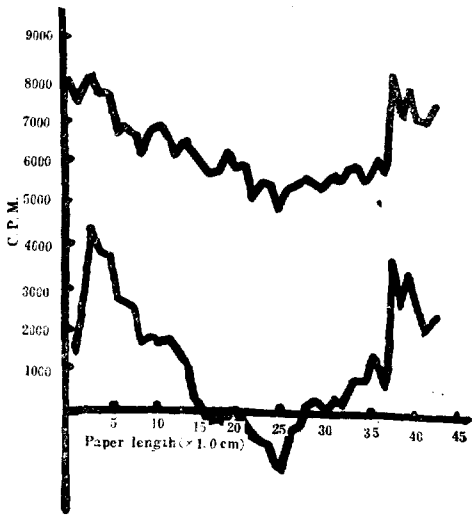


Fig 6. Alternative thickness calibrated method
C_6H_3 (CH_3) (NH_2) (I)
Solvent; 2-propanol: pyridine: water (8:1:1)
(a) Direct radiochromatogram
(b) Alternative thickness calibrated method.

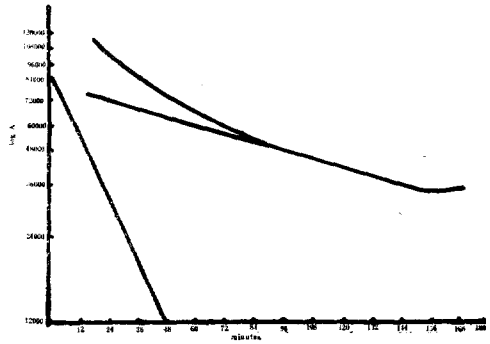


Fig 7. Decaying curve of the blank filter paper developed by the solvent (ethanol: pyridine: water).
Slope of the short lived content 11.2/5=2.24.

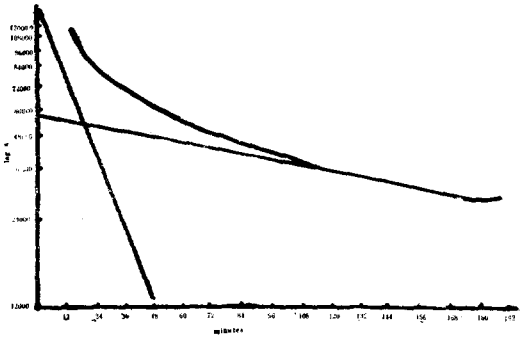


Fig 8. Decaying curve of the oil impregenated filter paper developed by the solvent (ethanol)
Slope of the short lived content 13.75/=2.74

Fig 9. Direct radiochromatogram.
$(CH_2ClCOOH)$
$A=T+S$, where A=total acitivity
T=thickness acitivity, S=sample activity

C. Thickness and activity decay calibration method.

Thickness calibration of the radiochromatogram was made by substracting the activity from that of direct radiochromatogram. The blank filter paper developed by the solvent without spotting the sample was irradiated and the activity decay curve was plotted in order to obtain the activity decay calibration factor. The decay curves of both ordinary phase and reversed phase were shown in Fig 7 and Fig 8. The time dependance of the decay of the activity was summarized in the table 1 and 2. The activity decay calibration was made in the following way: The activity calibration factor was multiplied to the activity of the thickness calibrated chromatogram and the activity was normalized to the initial section of the filterpaper. (The counting was conducted exactly after 50 minutes from the time of the irradiation. Therefore, the normalization was made to the time of 50 minutes after irradiation.) The chromatogram were shown in the Fig 9, 10, and 11.

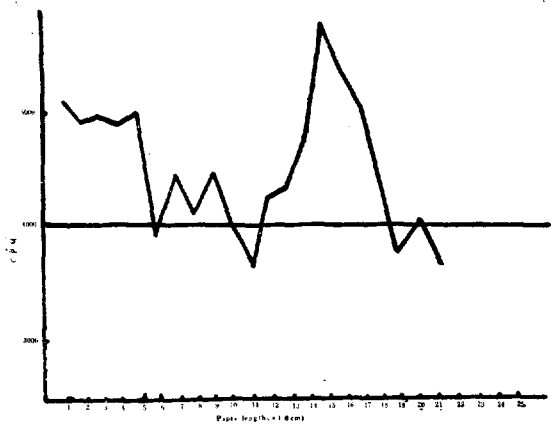


Fig 10. Thickness calibrated chromatogram ($CH_2CICOOH$ solvent: ethanol: pyridine; water).

$A=(T-Ta)+S$, where A=total activity
T=thickness activity of the filter paper
Ta=average thickness acivity
S=sample activity

Table1 1. Activity decay of the filter paper*.

time after Irradiation Minutes	activity of the filterpaper	relative decay ratio per 10 minutes. counts/minute
16	103.089	
26	85.483	82.92
36	74.083	86.66
46	65.218	88.03
56	57.535	88.22
66	52.755	91.99
76	47.988	90.36
86	44.202	92.73
96	41.262	93.35
106	38.651	93.67
116	36.587	94.65
126	35.039	95.77
136	33.117	94.51
146	31.431	94.91
156	30.470	96.94
166	29.799	97.80
176	28.849	96.81
186	28.145	97.59

* Irradiated under neutron flux of $1.5\times10^{12}n/cm^2$, sec using the pneumatic tube system of Triga Mark II. for 5 minutes.

Fig 11. Thickness and activity calibrated radiochromatogram ($CH_2CICOOH$). $A=(T-Ta)/c+S/c$
Where C is the activity calibration factor.

Table 2. Activity calibration factor of the filterpaper chromatogram irradiated and counted after 50 minutes from the irradiation. *

paper length (×1.0cm)	time after (1) irradiation (min.)	calibration factor (2)
1		88.02
2	52.6	88.17
3	55.2	88.22
4	57.8	86.17
5	60.4	84.12
6	63.0	82.07
7	65.6	80.89
8	68.2	79.32
9	70.8	77.75
10	73.4	76.18
11	76.0	73.18
12	78.6	74.61
13	81.2	71.73
14	83.8	70.28
15	86.4	68.83
16	89.0	67.42
17	91.6	66.03
18	94.2	64.64
19	96 8	63.25
20	99.4	62.30
21	10.0	61.35

(1) The activity decay of the irradiated filter paper was time dependent as shown in Fig 6. Therefore, it is necessary to note the time of the initial counting. If the time differ, more than 50 minutes, the factor should be recalculated from the decay curve and decay mode in the table 1.

* Irradiated under a neutron flux of $1.5\times10^{12}n/cm^2$, sec. using Triga Mark II for 5 minutes.

(2) The factor was calculated as follows:
Initial factor × factor of 10 minutes interval. (see Table 1.) The factor involved between ten minutes intervals was obtained by averaging.

Table 3. Activity decay of the filter paper* (Reversed phase).

time after irradiation minutes	count/minute	relative decay ratio per 10 minutes
15	113922	73.09
25	83273	87.15
35	72569	88.92
45	64523	91.01
55	58724	89.07
65	52306	94.33
75	49340	92.33
85	45840	92.91
95	43297	94.45
105	40727	94.95
115	38891	95.45
125	36947	95.00
135	35683	96.58
145	34812	97.56
155	33502	96.24
165	33140	98.92
175	31527	95.13
185	31652	100.40
195	30792	97.28

* The filter paper was irradiated under a neutron flux of $1.5 \times 10^{12} n/cm^2$, sec.

Table 4. Activity decay calibration factor.

paper length (x1.0cm)	time after irradiation (Min.)	calibration factor (%)
1	50.00	83.5
2	52.6	80.8
3	55.2	78.1
4	57.8	75.8
5	60.4	73.7
6	63.0	71.6
7	65.6	69.5
8	68.2	67.7
9	70.8	66.1
10	73.4	64.5
11	76.0	62.9
12	78.6	61.7
13	81.2	60.4
14	83.8	59.1
15	86.4	57.8
16	89.0	56.7
17	91.6	55.8
18	94.2	54.9
19	96.8	54.0
20	00.4	53.1
21	102.0	52.3

Table 5. An example of thickness and actvity calibrated ratiomatogram.

Sample: CH_2Cl COOH; Filter paper: Whatmann No. 1
Developing Solvent: Ethyl alcohol, pyridine, water (8:1:1) Irradiation: 5 minutes

paper length (×1.0 cm)	$T+S$	$(T-Ta)+S$	C %	$\{(T-Ta)+S\}/C$
1	77700*	50988*	0	50685*
2	70400	43186	88.17	48,980
3	70000	43599	88.22	49,420
4	66800	42815	86.17	48,800
5	65100	42099	84.12	50,046
6	58900	32439	82.07	39,046
7	61000	36754	80.89	44,207
8	57500	32694	79.32	41,331
9	59500	34710	77.95	44,643
10	55800	30561	76.18	40,158
11	50900	27264	74.61	36,542
12	55100	30142	73.18	42,556
13	55800	31638	71.73	43,300
14	54500	33431	70.28	49,568
15	69300	39419	68.83	57,271
16	54800	35898	67.42	53,246
17	59600	33335	66.03	50,484
18	52000	28293	64.64	43,782
19	36200	23806	63.25	37,640
20	36100	24354	62.30	40,000
21	32100	22356	61.35	36,440

* Plotting of this value was shown in Fig 9, 10, and 11.
T=thickness activity, Ta=blank filter paper activity
S=sample activity, C=activity calibration factor

Result and Discussion

Paper partion chromatography of organic halogen compounds, especially polyhalogenated compounds, were investigated. The usual techniques were applied for the case of simple chlorinated compounds, i. e., monochlorine on the one carbon atom (CH_2Cl COOH) and was able to obtain the spot on the filterpaper by coloring the developed spot by means of an ammoniacal solution of silver nitrate. Various solvents were applied in order to effect the efficient separation of the components. Ethyl alcohol, pyridine, water, 2-propanol, pyridine, water, ethyl alcohol, acetic acid, water; and butyl alcohol, benzene saturated by sodium carbonate solution were used as the developing solvent. Generally, the chlorine compounds were apparently developed on the paper at the paper length of 2-15 cm, but the iodo compounds were sometimes developed at higher position. Among the developing solvents ethyl alcohol system

was the best one for the chloro, iodo, and fluoro acids, but 2-propanol system was better for the case of halides. In the case of acetic acid mixed solvent the spot was more efficient than any others. However, organic halides were more efficiently developed when the filter paper was impregenated with liquid paraffin (a reversed phase) and developed with ethyl alcohol. The concentration of developed spot was larger than that of ordinary phase. This was quite natural when the solubility of the oily halides were considered [5] (see Fig 5). When the chlorination mixture of phenyl derivatives was developed on a reversed phased paper the spots were clearer than that of ordinary water phase and the R_f value was higher. [5] The analysis of the spot was easy for the case of monochlorinated compounds when the filterpaper was sprayed with ammoniacal or acidified silver nitrate solution and exposed under sunlight for a few minutes. Black to violet color was distinctly appeared. However, iodides gave a faint pink or violet color which was difficult to identify. For the case of poly-halogenated acids or alcohols such as C_3F_7COOH and $CCl_3C(OH)(CH_3)_2$ spots could not be identified by this technique. Probably the poly halogenated halogen atoms in the molecules are inert to be cleaved by silver nitrate solution.

Therefore, a series of experiments was conducted to develop a method of identifying these compounds by the paper partition chlomatography. In the previous research done by the author a separation of the components of chlorinated mixture of phenyl derivatives was achieved by applying a radiochromatographic technique. [5] The same technique was considered to solve the present problem. The chromatogram obtained was shown in the Fig 5 chromatograph 1, 2, 3, and 4. The activity induced by filter paper was generally low as experienced before (3), but the back ground activity was high in some area on the filter paper, which was significant enough to misidentify the small peak of a spot. The results was considered that the back ground induced by the reactor irradiation was mainly due to the filter paper composition itself which varies its activity depending on the thickness of the filter paper. Therefore, thickness calibration was attempted.

A filter paper was developed without spotting the sample by the same solvent used and it was irradiated. At the same time a filter paper without developing by the solvent was irradiated. The activity by the filter-papers were plotted against the time (Fig 7 and 8). In one case the activity was examined by counting the section of the paper at 2.6 minutes interval (Fig 6). From the activity measurement and plotting, it was observed that the activity induced by the filterpaper depends on the thickness of the filter paper and the time elasped after the irradiation. For the thickness, various parts of the Whatmann #1 filter paper were examined and the average of the filter paper was plotted against the paper length. The difference was generally within 2000 counts per minute at a gain of the counter (Fig 6).

To eliminate the thickness factor, two irradiations were made in one batch so that an error by time or geometry can be eliminated. Two irradiated filter papers were treated as described in the direct method and the activity of the each section was measured alternatively, i.e. the section of the sample filter paper was counted and the same section of the blank filterpaper was counted in the next and so on. The obtained activity was plotted against the paper length. The original radio chromatogram of the sample is shown in Fig 6-(a). The chromatogram indicates that the peak appeared at 5 and 38-40 paper section. However, there were many significant peaks at other area. Therefore, the activity of the blank filter paper was substracted from that of the sample in order to eliminate the activity of the filter paper from the chromatogram The difference in the acitvity thus obtained was plotted against paper length to give the chromatogram (b). There obtained a clear chromatogram of comparatively low filter paper background. However, this method was good for a long half lived radioisotope species with a high Rf value. Since the counting time was 2.6 minutes interval between two sections of the same filter paper, the activity of the sample of the spot was decayed rapidly at the high Rf value region when the radio isotope formed on the spot is of a short half-life and the concentration of the spot is low. For the case of a spot obtained at a low Rf value the calibration was not sufficient enough because the variation of the filter paper background at this area is significantly mixed with the sample peak.

For the decay of the filter paper activity, the decay curve was examined. Since the counting time was aro-

und 56-60 minutes from the initial counting of the first section of a filter paper to the last section of the filter paper, a considerable decrease in the activity of the filter paper background will be expected if the half life of the filter paper activity is of a short species. The decay mode of the filter paper was shown in the Fig 8, 9 and tables 1, 2. From the decay curve it was observed that the activity of the filter paper was composed of that of several species of nucleides contained in the irradiated paper. Therefore, it was difficult to find a general factor of calibrating the activity decay of the filter paper background. The decay varied largely as the time of counting was varied. In order to find a method of determinining the calibration factor of the activity decay, an approximation method was improvised. The relative decay ratio at 10 minutes interval was calculated, which was shown in table 3 and 4. The value was not linearly preportional and it varied significantly at the short time interval immediately after the time of the irradiation. A relative linear average was obtained at 50-106 minutes after the irradiation. Therefore these time intervals were considered as the counting period of the irradiated paper. A calibration factor was calculated as described in table 3 and 4. The calibration factor thus obtained was shown in table 3 and 4. With this activity decay calibration factor and the filterpaper thickness variation mode a activity and filterpaper thickness calibration method was made. Thus, the original activity of the irradiated filterpaper was;

$$A=T+S \qquad (1)$$

where, A=activity

T=activity of the filterpaper backgroud

S=activity of the spot

The activity of irradiated filter paper calibrated by filter paper thickness is as follows;

$$A=(T-Ta)+S \qquad (1)$$

where, A=activity

T=the filter paper background of the sample filter paper.

T_a=the filter paper bakground of the blank filter paper.

S=activity of the spot

The activity of the irradiated filter paper calibrated by activity and thickness calibrated is as follows;

$$A=\{(T-Ta)+S\}/C$$

where, A=activity

T, T_a and S are as above.

C=activity calibration factor.

The radio chromatogram obtained were shown in Fig 9, 10, and 11. There observed that the calibrated blank filter paper was generally low in its background except at 5-7 and 16-18 paper length (1.0 cm interval), whereas the same filter paper developed showed higher backgound at 7-14 paper length with remarkable activity drop at higher paper length sections. This was probably due to the solvent activation which remained in the paper tissue. It was, however, dualy eliminated from the background of the sample filter paper when the thickness calibration was made. As it is shown the chromatogram thus obtained was relatively low in its background and the spot peak was clear enough to identify. The example of the calibration was shown in the table 5. In some cases the activity drop at the higher paper length region was not eliminated even after the calibration, but this did not interfere the identification of the spot at lower, paper length region. The considerablly high activity of the initial section of the paper was observed in every cases, which was probably due to the sample remained undeveloped near at the spotting line.

The peaks of radio chromatogram was compared with the spot obtained by coloring method, which was shown in table 6. The spot and peak were generally matched each other at the same paper length region. In cases of thickness calibrated there were some high background peaks, whereas the activity and thickness calibrated case few foreign peaks were only observed. Surprisingly polyfluorinated acid and trichloro alcohol showed a distinct peak in radiochromatogram whereas the same compounds could not give spot by coloring technique. This method, therefore, seemed to be promising for the separation and identification of polyhalogenated compounds. The intensity of the spot was generally high in the case of reversed phase as expected, but the filterpaper background was high in this case even after calibration which was due to the variation in the thickness of the layer of coating material along the filter paper. However, the insoluble halides in a hydrophylic solvent was able to be identified better in the reversed phase because the intensity of the spot was high enough to surpass the filter paper background.

In conclusion the present method was valuable for the identification of organic halogen compounds by means of paper partition chromatography. Especially a polyhalogenated compounds, which is difficult to be identified by the usual paper chromatographic technique, can be identified efficiently. Further efforts are now under way to develop a quantitative radio chromatography of various halogen compounds. Because of the simplicity and convenience the manual counting of the filter paper strip was made in the present investigation for a qualitative purpose, but a scanning device will be helpful for both qualitative and quantitative works.

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Table 6. Qualitative spot analysis of radio paper chromatography of organic halogen compounds.

Sample	Phase	Solvent	Spot appeared* by conventional technique (cm) (O.1N $AgNO_3$ coloring)	Spot appeared* by Radiochromatography (cm)
$CH_2ClCOOH$	Ordinary	Ethanol: Pyridine: H_2O (8:1:1)	14	(a) 15 (3, 6, 9)
CCl_3-C(OH)-CH_3	〃	〃	does not appear	15(a) (3, 5, 19,)
C_3F_7COOH	〃	〃	〃	15(a) (5, 11,)
H_2N-C$_6$H$_3$(-I)-CH_3	〃	Ethanol: HOAc: H_2O (8:1:1)	21	19, 21(b) (2, 5)
$CH_2ClCOOH$	〃	2-propanol : pyridine : water (8:1:1)	11.5	(a) 11(9)
$CH_2ClCOOC_2H_5$	〃	〃	12.5	(b) 12.5
CCl_3-C(OH)-CH_3	〃	〃	does not appear	(a) 11(8)
C_3F_7COOH	〃	〃	〃	11(a)
$CH_2ClCOOC_2H_5$	reversed	C_2H_5OH	12	12(16)(b)
o-iodo toluene	〃	〃	14	14.5 (7.5, 12.5)

* Spot position was expressed by the paper length from the spotting line. The distance from the center of the spot to the spottng line was measured.

(a) Thickness and activity calibration were made. The values in the parantheses denote the filter paper background, but they were minor as compared to the uncalibrated chromatogram. (See Fig 1)

(b) Alternate thickness calibration or direct methods were used. Generally, the values in the parenthes, which denote the presence of impurities of filter paper background, were significant enough to misinterprete the genuine spot peaks. (See Fig 5, 6)