# FINSKA KEMISTSAMFUNDETS MEDDELANDEN

# SUOMEN KEMISTISEURAN TIEDONANTOJA

REDAKTÖR — TOIMITTAJA

**Harald Nyberg**


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# Studies of Dehydrohalogenation Catalysts for the Manufacture of Vinylchloride from 1,2-Dichloroethane


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## Summary

As introduction a survey is given of the literature concerning catalysts for the fission of 1,2-dichloroethane to vinyl chloride and hydrogen chloride.

Introductive experiments, where only the formation of hydrogen chloride was determined, showed that among others the following substances catalyze the dehydrohalogenation of 1,2-dichloroethane: Fe-tube at $> 500°$, $CaCl_2$ at 500°, $V_2O_5$, blue silica gel, bleaching earths Terrana, Fulmont, Tonsil AC, Surrey Powder, Nolek NZ at 340°; phosphotungstic acid at 340 and 255°; silica gel impregnated with the following substances: $FeCl_2$, $MnSO_4$, $AlCl_3$, $Cr_2(SO_4)_3$, $CdSO_4$, $Th(SO_4)_2$, $ThCl_4$, $Zr(SO_4)_2$, $TiO_2$, $TiSO_4$, $H_2TIO_3$, $VSO_4$, $CoCl_2$, $ZnCl_2$ and $ZnSO_4$ at 255°.

At experiments, where eventually formed vinyl chloride was isolated in a cold trap, it was found that practically no vinyl chloride was formed with zinc salts on silicagel or with Terrana bleaching earth catalyst. The zinc salts formed only small amounts of gaseous products, but the bleaching earth Terrana

at 300 or 310° gave considerable amounts of ethylene, about one mole for four or five moles of HCl (compare (1)). No acetylene or chlorine could be detected.

Experiments with active carbon catalysts gave yields of up to 99.5 mole % of HCl and 88 mole % of vinyl chloride at about 300°. Also here some ethylene was formed, but only in amounts less than one mole %. About 2—5 mole % of substance, calculated as vinyl chloride, are deposited on the catalyst. Only a slight fouling of the catalyst could, however, be observed. Impregnation of the carbon with barium chloride gives only a slight increase of the once through yields of vinyl chloride and hydrogen chloride.

### *Earlier investigations*

Vinyl chloride, the important starting material for vinyl plastics, is manufactured by addition of hydrogen chloride to acetylene (reaction I), or by dehydrohalogenation of 1,2-dichloroethane (ethylene dichloride) (reaction II). Other preparative

$$C_2H_2 + HCl = CH_2{:}CHCl \quad (I) \qquad C_2H_4Cl_2 = CH_2{:}CHCl + HCl \quad (II)$$

methods proposed are the substituting chlorination of ethylene (reaction III) [2] or of ethane [3], oxidation of ethyl chloride by air with copper oxide catalyst (reaction IV) [4], or conversion of a mixture of 1,1-dichloroethane and ethylene with a calcium sulfate catalyst at 260° [5].

$$C_2H_4 + Cl_2 = CH_2{:}CHCl + HCl \quad (III)$$

$$C_2H_5Cl + O = C_2H_3Cl + H_2O \quad (IV$$

A ferric chloride catalyst on carriers such as Fullers earth, silica gel or pumice has been used for the chlorination of ethylene to vinyl chloride [6].

Reaction I has been much studied [7]. It is usually carried out with the aid of mercury containing catalysts, frequently mercuric chloride. The use of mercury salts for this purpose has been known at least since 1914 [8]. Carbon is usually used as catalyst carrier, but also pumice, silica gel, polymethyl acrylamide, polymers of perlon or nylon type, and a polyamine ion exchange resin have been recommended for this purpose [9]. The suitable temperature is between 100 and 250°C [10,39] but it has been stated that it should be possible to use temperatures lower than 100° with mercuric chloride or cadmium chloride catalysts [11], or with liquid copper containing catalysts [12].

Other catalysts used at reaction I are $HgCl_2$ together with $CaCl_2$ and $BaCl_2$ on charcoal or silica gel as carrier [13], $HgCl_2$ + $Al(OH)_3$ [14], $HgCl_2$ on $Al_2O_3$ [15], heavy metal fluorides or fluosilicates, e.g. $HgF_2$ or zinc fluosilicate on active carbon [16], chlorovinyl mercuric chloride and the corresponding bis compound [17], arsene halovinyl compounds [18], nitriles such as $CH_3CN$,$C_6H_5CN$ etc. in ether or benzene [19], $HgCl_2$ on active carbon suspended in paraffin oil, $C_2HCl_5$ or other diluent [20], $HgCl_2$ + titan or



thorium oxide [21], $HgCl_2$ + $ThCl_4$ on active carbon [22], a complex salt of mercury and cerium halides [23], mercury vanadate [24], mercury vapor, $HgCl$ [25], gold halides on charcoal [26], mercury and copper halides on active carbon, silica gel or pumice [27], $FeCl_3$ and $HgCl_2$ suspended in an inert solvent [28], $Hg_3(PO_4)_2$ or $Hg_3(As_4)_2$ on carbon activated with phosphoric, sulfuric or perchloric acid (Fe and Zn in the catalyst form high boiling byproducts) [30], copper salts on charcoal [31] or in water solution [32]. With copper salts as catalysts vinyl chloride is formed in strongly acidic, but vinyl acetylene in neutral solution [33] (compare) [34].

Passing the gas mixture over $NiCl_2$ [35,36] or a mixture of kieselguhr, $FeCl_3$, $HgCl_2$, $CuCl_2$ and $H_2O$ [37] before it comes into contact with the Hg-catalyst, or saturating the gas with water vapor [38] is said to be of advantage. The use of superpressure (3 athm. [36]), or a fluidized $HgCl_2$-charcoal catalyst [37], or of ammonium chloride instead of hydrogen chloride [38] has been proposed. A $HgCl_2$ on alumina-catalyst is claimed to give conversions of acetylene to vinyl chloride of 96—98 mole % originally, 13 % only after poisoning with hydrogen sulfide at 80°, and 98 % again after treating the poisoned material for 1 hour with chlorine at 100° and then for another hour with hydrogen chloride to desorb chlorine [39]. In some cases the addition of water to acetylene giving acetaldehyde is obviously a complicating reaction [34].

The dehydrohalogenation of 1,2-dichloro ethane is carried out industrially on a small scale by treating this chlorine compound with sodium hydroxide (reaction V) [39], or with calcium hydroxide under about 8—12 athm. pressure [44]. Presence of substances with alcoholic or phenolic [45] hydroxyl

$$C_2H_4Cl_2 + NaOH = C_2H_3Cl + NaCl + H_2O \quad (V)$$

groups, for instance methanol [46], ethanol [47,48], butanol, amyl alcohol, cyclohexanol [49], polyhydric alcohols [50], tetraethylene glycol [51], ethylene glycol or its monoethers [47] seems to be of advantage at this reaction. The use of sodium carbonate [53] or of quaternary ammonium bases $R_4NOH$ [54] instead of sodium hydroxide has been proposed.

A fission of dichloroethane to vinyl chloride and hydrogen chloride according to (II) would be more advantageous than reaction, V, as free hydrogen chloride is more valuable than sodium chloride, among other things because it can be utilized for reaction I. Several inventors have in fact devised processes combining reactions I and II. In some of these processes both reactions are carried out simultaneously in one step, in others reactions I and II go on separately in different parts of the apparatus and usually with different catalysts [55-60]. As catalysts for a simultaneous reaction according to (I) and (II) charcoal impregnated with $BaCl_2$, $SnCl_4$, $FeCl_3$, $HgCl$ or $ZnCl_2$ [61] or also with

$BiCl_3$, $HgCl_2$, and $CdCl_2$ [62], with $BaCl_2$ and $HgCl_2$ [63] or HgCl and KCl [4] have been recommended.

In a country like Finland, where dichloroethane can be made easily from alcohol obtained in the sulfite cellulose industry the fission of dichloroethane according to reaction II must obviously be the fundamental reaction in the manufacture of vinyl chloride.

According to literature it is usually carried out by simple cracking, leading dichloroethane vapors through heated empty tubes [57,58,65-69] or tubes filled with chamotte [60], alumina [70], pumice [71] or, preferably, smooth surfaced pebble gravel [72] at about 400—675° or according to an early work [73], even 800—1 000°C. The use of a fluidized bed of fine sand at 510—600° [74] or of fluidized bauxite at 350° [75] has been proposed, and also ceramic packing or sand heated by reacting hydrogen with chlorine [76].

The once through yield is said to be 70 mole %, which can be increased to 97—98° by recycling [77]. The high temperatures used present some technical and heat economical disadvantages. In addition, it is stated [78], that the pyrolysis of 1,2-dichloroethane, if carried out at temperatures above 540°, gives on fractionation a vinyl chloride which has a very low rate of polymerization, probably owing to the formation of byproducts impeding the polymerization. One of them is believed to be vinylacetylene, $CH_2$:CH.C:CH,B.P. + 3°, which, however, can be removed by careful fractionation. According to [79], the impeding byproducts can be removed by treating the vinyl chloride with conc. sulfuric acid or with chlorine. The use of catalysts which would make it possible to carry out the reaction at lower temperatures, would obviously be of interest. Investigations of catalysts for this purpose have in fact been carried out, for instance using active carbon as catalyst at 230—250° [80], at 325—350° [81,82], at 340—500° [83], at 435—500° [84], or 480—520° [79], carbon impregnated with $BaCl_2$ or $CuCl_2$ [85], or with $BaCl_2$ and $HgCl_2$ [86], a quartz tube packed alternately with glass rings and active carbon [87], fluidized carbon catalyst containing $HgCl_2$ [88], reduced Cu or Fe on fire brick [89], $CCl_4$ [90], Cl,Br,$SO_2Cl_2$, oxygen or air at 300—345° [91]. The use of glowing platinum wire in a ketene lamp [92] or liquid phase dehydrohalogenation with an immersed heating element of 800—1 100° [93] has also been proposed. Anhydrous aluminium chloride splits off one mole of hydrogen chloride from 1,2-dichloroethane already at 45—55°, but the vinyl chloride formed is immediately polymerized [94]. A 80—85° the reaction involves a loss of two molecules of hydrogen chloride, yielding acetylene [95].

The dehydrogalohenation of 1,2-dichloroethane is said to proceed according to the following chain mechanism [96], (compare [97]):



$$C_2H_4Cl_2 \rightarrow C_2H_4Cl^{\times} + Cl^{\times}$$
$$Cl^{\times} + C_2H_4Cl_2 \rightarrow C_2H_3Cl_2{}^{\times} + HCl$$
$$C_2H_3Cl_2{}^{\times} \rightarrow C_2H_3Cl + Cl^{\times}$$
$$Cl^{\times} + C_2H_4Cl^{\times} \rightarrow C_2H_3Cl + HCl$$

As complicating reactions, which can decrease the yield of vinyl chloride, the following are reported:

$$C_2H_3Cl + HCl = CH_3CHCl_2 \qquad \text{(VI)}\ [94]$$
$$C_2H_3Cl = C_2H_2 + HCl \qquad \text{(VII)}\ [57,66,67,73].$$

The reverse reaction to (VI), the formation of vinyl chloride from ethylidene chloride and perhaps also from other chloroethanes, is said to be catalyzed by ferric chloride [99].

Combinations of the reactions II and V have been proposed, using Cl, Br or oxygen as catalysts at 360 [67,87]. Further,[101] combines reaction II with the addition of chlorine to ethylene in the same reaction chamber. By combining the endothermic dehydrohalogenation reaction with the exothermic halogenation the reaction is effected at 300—400°, the decomposition of 1,2-dichloro ethane being catalyzed by the chlorine atoms or other active radicals, as oxygen, which is added by aeration of the dichloroethane. The yield of vinyl chloride is claimed to be 83—87 % [102]. combines dehydrohalogenation of acetylene tetrachloride with addition of hydrogen chloride to acetylene, obtaining vinyl chloride and trichloro ethylene. Active carbon impregnated with $BaCl_2$, $BiCl_3$, $FeCl_3$, $CdCl_2$, $HgCl_2$ or $ZnCl_2$ is used as catalyst and the reaction temperature is about 250°.

## Own investigations

*Experiments with vapors catalysts, i. a. zinc salts and bleaching earths.*

The purpose of the present work was to study the use of catalysts at the dehydrohalogenation of 1,2-dichloroethane.

The usual technique at earlier experiments for the same purpose seems to have been to lead dichloroethane vapor over the heated catalyst, absorb the gaseous reaction products in water and titrate the hydrogen chloride formed. Doraswany and Pay [84] are of opinion that such a procedure gives a fairly true measure of the formation of vinyl chloride, as only insignificant amounts of byproducts are formed, of which they name acetylene only. According to Barton et al., the formation of acetylene from[103] 1,2-dichloroethane in empty tubes should be negligible at temperatures up to 485°. Therefore, the technique referred to was at first employed also at the present work. The first experiments were carried out using the simple apparatus shown in

fig. 1. Usually, one ml of dichloroethane was introduced with a speed of about one drop in 5 seconds in the Claisen flask C, where it was vaporized. The vapors passed with a current of nitrogen of about 2 bubbles per second through the tube U containing the catalyst, usually 7 g, and granulated and sieved to the dimension 1—5 mm. The hydrogen chloride formed was absorbed in water in the scrubbing bottle W, where it was titrated after the experiment with 0.1 N NaOH, with methyl orange as indicator.

The results of a number of experiments with this procedure carried out by Mrs Maja Vahtila and Mr Osmo Nissilä are related in the tables 1—7 and the lists of experiments with less active substances connected to these tables. These show that several simple substances are strong catalysts for the splitting off of hydrogen chloride from dichloroethane, among these phosphotungstic acid, earlier known as a decarboxylation catalyst for resin acids [104], and further bleaching earths such as Terrana extra, Fulmont and Tonsil AC. Blue silica gel for drying purposes was found to catalyze rather feebly, but, however, considerably stronger than a colorless silica gel. As the color of blue silica gel seems to be due to impregnation with cobalt salts, this led

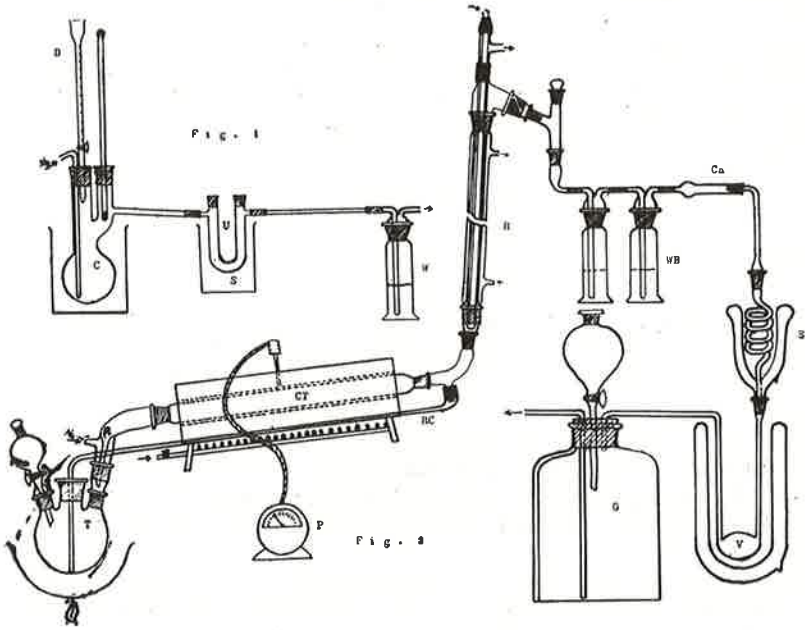


*Fig. 1.* Apparatus for preliminary dehydrohalogenation of 1 ml dichloroethane.
*Fig. 2.* Apparatus for dehydrohalogenation of about 25—100 g dichloroethane.



Table 1. *Experiments with 1 ml = 12.7 millimoles of $C_2H_4Cl_2$ at about 500° in straight tubes.*

| Exp. No | Tube material | Catalyst | t°C | Developed HCl, millimoles |
|---|---|---|---|---|
| 1 | Iron | — | > 500° [1] | 13.8 |
| 2 | Glass | — | ca 500° | 0.89 |
| 3 | » | — | 340° | 0.03 |
| 4 | » | $CaCl_2$ | ca 500° | 7.87 |
| 5 | » | $CaCl_2$ | ca 340° | 0.12 |

[1] Red hot.

Tabe 2. *Experiments with 1+1 ml $C_2H_4Cl_2$ at 340°.* U-tube of glass of diameter 13 mm, fig. 1. Catalyst 1—5 mm pellets, layer 70 mm long.

| Exp. | Catalyst | Developed HCl, millimoles I | II | Remarks |
|---|---|---|---|---|
| 20 | $V_2O_5$ | 9.67 | 7.15 | |
| | Bleching Earths: | | | |
| 27 | Terrana | 12.35 [1] | 2.71 | Darkened |
| 28 | Fulmont | 9.20 | 7.23 | » |
| 29 | Tonsil AC | 8.57 | 3.23 | » cat. without $C_2H_4Cl_2$: 0.35 |
| 30 | Surrey Powder | 3.79 | 5.53 | » |
| 31 | Nobek NZ | 2.30 | — | |
| 32 | Silicagel (blue) | 9.26 | 4.25 | |
| 33 | Magnesol | 0.86 | 2.07 | |

[1] At 300°, with 2 g of catalyst, 1.43.

1 ml of $C_2H_4Cl_2$ gave 0.1—1 millimoles of HCl at 340° with the following catalysts: $CaCl_2$ 0.12, porcelain splittings 0.11, Fe-powder practic. 0.20, $Al_2O_3$ 0.13, $BaCl_2$ 0.11, $ZnCl_2$ 0.15 (cat. only 0.03).

Less than 0.1 millimoles HCl gave at 340°C: Cu-netting, pure Fe-powder, BaO, $P_2O_5$, celite (kieselguhr), magnesol.

Table 3. *Experiments with bleaching earth Tonsil AC or gas adsorption Carbon (Merck) and 1 ml of $C_2H_4Cl_2$ at various temperatures.* App. and cat. as in table 2.

| Exp. No | Catalyst | °C | Millimoles HCl | Remarks |
|---|---|---|---|---|
| 8 | Tonsil AC | 200 | 0.02 | White |
| 9 | » » | 224 | 0.06 | » |
| 10 | » » | 235 | 0.08 | Grey |
| 11 | » » | 255 | 0.38 | Dark grey |
| 12 | » » | 285 | 0.73 | Dark |
| 13 | » » | 340 | 8.57 | Black |
| 112 | Active C (Merck) | 340 | 4.51 [1] | |
| 113 | » | 280 | 0.25 | |
| 114 | » | 255 | 0.05 | |

Dowex cation exchanger gave no HCl at 100° or even at 255°.

[1] With a 2nd portion of 1.0 ml $C_2H_4Cl_2$: 5.30.

Table 4. *Experiments with 1 + 1 ml of $C_2H_4Cl_2$ at 285°C.*
App. and catalyst as reported in table 2.

| Exp. No | Catalyst: Carrier | Catalyst: Added sbst. | Millimoles HCl I | Millimoles HCl II | Remarks |
|---|---|---|---|---|---|
| 41 | Terrana | — | 0.80 | 0.65 | Darkened |
| 51 | | Phosphotungstic acid, | | | |
| | | » 1% | 1.38 | 0.81 | |
| 52 | » | » , 10% | 1.64 | 1.36 | |
| 53 | » | » , 50% | 3.33 | 0.79 | |
| 54 | Active C | » , 10% | 0.45 | 0.47 | |

Restricted amounts of HCl gave at 285°C the following catalysts: Fulmont 0.57 + 0.41, Tonsil AC 0.73 + 0.24, Nobek NZ 0.17, phosphotungstic acid 0.90 + 0.55.

Amounts of HCl < 0.1 millimole gave at 285°: CoO, $P_2O_5$, $V_2O_5$, kaoline, phosphomolybdic acid, $H_3PO_4$, $MoO_3$, $CrO_3$.

Table 5. *Experiments with silica gel and some additional substances at 255°C with 1 or 1 + 1 ml of $C_2H_4Cl_2$.*
App. and catalysts as in table 2. Amount of catalyst 7.0 g at all experiments designed N.

| Exp. No | Carrier | Added sbst. | millimoles HCl I | HCl II [1] | Catalyst only | Remarks |
|---|---|---|---|---|---|---|
| 89 | Silica gel, colorless | — | 0.03 | — | | |
| 88 | » » blue | — | 0.28 | 0.33 | | |
| 62 | » » » | 10 % $FeCl_2$ | 2.18 | 2.06 | 0.22 | |
| 63 | » » colorless | —»— | 1.27 | 1.73 | 0.02 | |
| 64 | » » » | —»— | 0.93 | 0.83 | 0.92 | |
| 20 N | » » » | 10 % $FeSO_4$ | 0.85 | — | — | Darkened |
| 22 N | » » » | 10 % $MnSO_4$ | 1.58 | — | — | » |
| 14 N | » » blue | 10 % $KAlSO_4$ [2] | 0.66 | — | 0.05 | » |
| 26 N | » » » | 10 % $AlCl_3$ | 4.14 | — | 2.00 | » |
| 15 N | » » » | 10 % $Cr_2(SO_4)_3$ | 1.49 | — | 0.22 | » |
| 16 N | » » » | 10 % $KCr(SO_4)_2$ | 0.62 | — | 0.09 | » |
| 23 N | » » » | 10 % $CdSO_4$ | 3.78 | — | — | » |
| 33 N | » » » | 10 % $Th(SO_4)_2$ | 1.02 | — | 0.09 | » |
| 34 N | » » » | 10 % $ThCl_4$ | 1.03 | — | 0.67 | » |
| 36 N | » » » | 10 % $Zr(SO_4)_3$ | 1.16 | — | 0.08 | » |
| 38 N | » » » | 10 % $TiO_2$ | 1.31 | — | — | » |
| 39 N | » » » | 10 % $TiSO_4$ | 1.33 | — | 0.03 | » |
| 40 N | » » » | 10 % $H_2TiO_3$ [3] | 1.33 | — | — | » |
| 41 N | » » » | 10 % $VSO_4$ | 1.14 | — | — | » |

[1] A dash in this column means that no experiment was carried out.
[2] An experiment with $Al_2(SO_4)_3$ gave the result that the catalyst sublimed.
[3] In this case only 5 g of catalyst was used.



Table 6. *Experiments with phosphotungstic acid at 255°C.* 1+1 ml $C_2H_4Cl_2$.
App. as in table 2. Phosphotungstic acid designed in the table as PW-acid.

| Exp. No | Carrier | Added sbst. | Millimoles I | HCl II | Remarks |
|---|---|---|---|---|---|
| 91 | — | PW-acid pro anal. | 1.11 [4] | 0.76 | |
| 95 | — | » » » | 0.91 [1] | 0.35 | |
| 92 | — | Same, after standing 22 days | 0.37 | — | |
| 93 | — | Same, calcined | 0.02 | — | |
| 94 | — | » practical | 0.09 | — | |
| 96 | — | PW-acid, fresh prep. | 0.42 | 0.18 | |
| 98 | — | » + HCl conc. | 0.38 | 0.15 | |
| 99 | — | » + 2% phosphomolybdic acid | 0.24 | 0.33 | |
| 100 | — | PW-acid + $HNO_3$ conc. | 0.40 | 0.34 | |
| 84 | Terrana | — | 0.28 | 0.28 | |
| 103 | » | 1 % PW-acid pro anal. | 0.38 | 0.39 | Darkened |
| 104 | » | 10 » » » » | 0.59 | 0.46 | » |
| 105 | » | 50 » » » » | 2.63 | 1.52 | » |
| 106 | » | 50 » » + phosphomolybdic acid [2] | 2.32 | 1.09 | » |
| 107 | Silica gel, blue | 50 » PW-acid pro anal. | 2.78 | 1.05 | » |
| 108 | Silicic acid [3] | 50 » » » » | 0.47 | 0.23 | |
| 109 | Celite (kieselguhr) | 10 » » » » | 0.02 | — | |
| 111 | China splits | 10 » » » » | 0.05 | — | |
| 110 | $V_2O_5$ | 50 » » » » | 0.71 | 0.45 | |

[1] Another preparate than that used at exp. 91.
[2] 2 % of PMo-acid in PW-acid, as in exp. 99.
[3] Prepared from $Na_2SiO_3$-solution with HCl.
[4] PW-acid at 200°C gave 0.02 millimoles of HCl.

to the investigation of silica gel impregnated with various salts by adding the salt in alcoholic or water solution to the silica gel and then evaporating the mixture to dryness on a water bath. It was soon found out that a considerable number of substances, among these $CoCl_2$, $FeCl_2$ and especially $ZnCl_2$ or $ZnSO_4$, when impregnated on silica gel as carrier gave catalysts, splitting off hydrogen chloride from dichloroethane very strongly.

Experiments with larger amounts of dichloroethane were carried out with the apparatus shown in fig. 2. The reaction was accomplished in a slow current of nitrogen in the gas heated catalyst tube CT of Pyrex glass, length 750, inner cross section 20 mm. The length of the catalyst layer was 170—200 mm. The unreacted dichloroethane was condensed in the vertical reflux condenser R and recycled to the tubulated flask T, where it was vaporized in an oil bath kept at about 120—150°. A current of nitrogen was led through the apparatus at the beginning and at the end of the experiment (before and after the boiling of dichloroethane). The hydrogen chloride formed was dissolved

Table 7. *Experiments with silica gel and various concentrations of $CoCl_2$ and $ZnSO_4$ at 255°.* 1+1 ml $C_2H_4Cl_2$. App. and catalysts as in table 2.

| Ex Nop. | Catalyst Carrier | Added sbst. | Millimoles I | HCl II | Catalyst only | Remarks |
|---|---|---|---|---|---|---|
| 66 | Silica gel, colorless | 1 % $CoCl_2$ | 0.13 | — | 0 | |
| 67 | » » » | 10 » » | 0.73 | 0.63 | 0.48 | |
| 68 | » » » | 20 » » | 1.07 | 0.80 | 0.13 | |
| 69 | » » » | 50 » » | 1.63 | 1.52 | 0.38 | |
| 70 | » » » | 70 » » | 1.11 | 0.75 | 0.68 | |
| 55 | — | $CoCl_2$ | 0.01 | — | — | |
| 74 | Silica gel, colorless | 10 » $ZnSO_4$ | 1.12 | 1.02 | — | Darkened |
| 1 N | Silicagel, blue | 1 » » | 1.64 | — | 0.18 | » |
| 2 N | » » | 10 » » | 5.01 | — | 0.08 | » |
| 3 N | » » | 20 » » | 6.58 | 6.48 [1] | 0.16 | » |
| 4 N | » » | 30 » » | 8.63 | — | 0.56 | » |
| 5 N | — | » | 0.13 | — | — | » |
| 6 N | Silica gel, colorless | 10 » » | 1.51 [2] | — | — | » |
| 7 N | » » » | 50 » » | 2.88 | — | — | » |
| 30 N | » » blue | 10 » » + 10 % $FeCl_2$ | 3.38 | — | 2.41 | » |

[1] The 3rd ml of $C_2H_4Cl_2$ gave 5.42, the 4th 5.18 millimoles of HCl.
[2] Terrana + 10 % $ZnSO_4$ gave 3.06.

Small amounts of HCl at 255°C gave the following catalysts: Tonsil AC 0.38 + 1.5, the same + 50 % of copper acetate 0.27 + 0.13, silica gel colorless + 10 % of the following salts: $MnCl_2$ 0.49 + 0.95, catalyst only 0.05; $NiCl_2$ 0.33 + 0.33, cat. only 0.05; $SnCl_2$ 0.18, $CoSO_4$ 0.86; silica gel blue + 10 % of the following substances: $MgSO_4$ 0.64, cat. only 0.16; Zinc acetate 0.35; $HgCl_2$ 0.44, cat. only 0.22, $Li_2SO_4$ 0.31, $ZrOCl_2$ 0.65, cat. only 0.48; cerium phosphate 0.58 ml, cerium sulfate 0.31, cerium oxide 0.11 ml, HCl-extract of lantanide concentrate 0.43, cat. only 0.55; corresp. $H_2SO_4$-extract 0.56, cat. only 0.31 ml.

Amounts of HCl < 0.1 millimole gave at 255°C: $FeCl_3$ ($FeCl_3$ without $C_2H_4Cl_2$ gave 36.7), Tonsil AC + $NH_4VO_3$, silicic acid prepared by precipitation of $Na_2SiO_3$-solution with HCl, or by the method of Nijkamp (105), or according to (106) using 50% $Al_2(SO_4)_2$ or 50% $CoCl_2$; Surrey Powder, $UO_3$, colorless silica gel + $Fe(OH_3)$; colorles ssilica gel + 10 % of cobaltacetate, $Co(NO_2)_3$, copper acetate, $Hg_2Cl_2$, $CaCl_2$, $BaCl_2$, or $PbCl_2$; blue silica gel + 10% $ZnCrO_4$, ZnO, $ZnCO_3$, zinc borate, $Tl_2SO_4$, $Zr(NO_3)_4$, ceriumoxalate or nitrate, or lantanide concentrate, $AlPO_4$, anion exchanger Permutit ES, and freshly prepared phenol-formaldehyde resin.

and later titrated in the scrubber flasks WB, and the vinyl chloride formed condensed in the glass vessel V by means of a cold trap containing solid carbon dioxide in ethanol. The non-condensed gases were collected in the gasometer flask G over concentrated sodium chloride solution. The results are reported in the tables 8—11. They comprise experiments with three types of catalyst, namely 1) zinc sulfate or chloride on silica gel, 2) bleaching earth Terrana extra, and 3) active carbon, which at two experiments (E 10, table 8, and T 9, table 10)



Table 8. *Experiments with 25 g = 0.253 mole of $C_2H_4Cl_2$.* App. fig. 2.

| Exp. No | | °C | Catalyst Carrier | Added sbst. | g [1] | Subst. condensed in V, g | Increase of weight of catalyst, g | Unreacted substance in T | HCl in WB moles |
|---|---|---|---|---|---|---|---|---|---|
| D | 1 | 255 [4] | Silicagel, blue | 20 % $ZnSO_4$ | 40 | 0.55 | 1.0 | 8.6 | 0.162 [2] |
| D | 2 | 300 [4] | Terrana | — | 20 | 0 | 0.7 | 13.0 | 0.130 [3] |
| D | 3 | 255 | Silica gel, blue | 20 % $ZnSO_4$ | 40 | — [5 6] | 0.4 | 21.2 | 0.044 |
| D | 4 | 300 | Terrana | | 30 | — [5 7] | 1.3 | 8.6 | 0.214 [3] |
| D | 5 | 335 | Gas absorpt. carbon (Merck) | | 25 | — [5 8] | 1.5 | 3.0 | 0.205 [9] |
| D | 6 | 255° | Silica gel, blue | — | 40 | 0 | — | — | — [10] |
| D | 7 | 285—330 | » » » | 10 % $ZnCl_2$ | | Ab. 0.2 | 0.5 | 11.5 | 0.191 [3] |
| D | 9 | 335 | Terrana | — | 30 | Ab. 0.2 [12] | 1.7 | 3.1 | 0.285 [11] |
| D | 10 | 335 | Gas absorpt. Carbon (Merck) | | 25 | 13.3 [13] | 1.4 | 3.0 | 0.117 [9] |

[1] Total weight of carrier and added salt.
[2] Reaction fast to beginwith but slowed down very soon.
[3] Reaction slow.
[4] Without current of $N_2$.
[5] In this case the condensing vessels S and V were left out.
[6] The gas collected in G had a volume of 1500 ml at about 20°C and contained 95.2 % $N_2$, 1.7 % $O_2$, 0.7 % $CO_2$, unsaturated compounds (olefines etc. dissolving in fuming sulfuric acid) 1.6 %, CO 0.4 %, $CH_4$ 0.4 %, but no $H_2$.
[7] The gas in G had a volume of 2500 ml and contained 46.0 % $N_2$, 1.3 % $O_2$, unsaturated comp. 46.1 %, $CO_2$ 2.7 %, CO 3.3 %, and $CH_4$ 0.6 %.
[8] Analysis of gas, 4750 ml in all (two gasometer vessels G were used), calculated as nitrogen free: $O_2$ 1.0 % unsaturated 79.3 %, $CO_2$ 12.7 %, CO 1.8 %.
[9] Reaction fast.
[10] Practically no reaction.
[11] Reaction faster than at exp. D 4.
[12] The gas in the two vessels G used had a total volume of 3950 ml at about 20°C and contained, calculated as $N_2$ free: $O_2$ 1.0 % unsaturated comp. 79.0 %, $CO_2$ 6.0 %, CO 2.5 %. The gas did not contain any acetylene (testing with ammonia-cuprochloride according to (108) was negative, comparing test with acetylene was positive).
[13] Of this amount 11.5 g evaporated fast at room temperature.

Table 9. *Experiments with 25—150 g of $C_2H_4Cl_2$.* App. fig. 2.

| Exp. No. | °C | Catalyst Kind | g | $C_2H_4Cl_2$ g | Yield of vinyl chloride Sbst. in V g | % of theoretical [4] | Increase of weight of catalyst | Unreacted substance in T | Yield of HCl moles % of theoret. | | Remarks 255°C at the beginning, reaction then slower. Faster at 285° |
|---|---|---|---|---|---|---|---|---|---|---|---|
| E 1 | 285 | Gas absorpt. C (Merck) | 25 | 25 | 10.5 | 75.6 | 1.0 | 3.0 | 0.192 | 86.4 | |
| E 2 | 255 | » | 25 | 25 | 8.8 | 68.0 | 4.7 | 4.5 | 0.158 | 76.3 | Reaction very slow |
| E 3 | 285 | » | 25 | 25 | 9.55 | 67.2 | 1.15 | 2.5 | 0.206 | 90.6 | |
| E 4 | 310 | » | 25 | 25 | 9.3 | 66.2 | 1.3 | 2.75 | 0.208 | 92.5 | |
| E 5 | 285 | » | 25 | 3× 25 | 10.5+14.8 +13.2=38.5 | 86.1 | 1.4 | 4.2 | 0.708 | 99.0 | |
| E 6 | 285—310 | » | 25 | 2×125 | 61.4+58.6= 120.0 g | 77.3 | 3.5 | 4.1 | 2.15 | 86.5 | 285° for first 125 g, 310° for second |
| E 7 | 310 | » | 25 | 125 | 65.8 | 85.7 | 2.25 | 3.35 | 1.19 | 96.8 | Catalyst from E 6 |
| E 8 [1] | 285 | » | 25 | 25 | 2.55 | 18.7 | 0.15 | 3.4 | 0.076 | 34.8 | |
| E 9 [1] | 285 | » | 25 | 25 | 2.3 | 16.5 | 0 | 2.9 | 0.068 | 30.4 | Catalyst from E 7 |
| E 10 | 285 | 11+10% of P—W-acid | 25 | 25 | 8.75 | 64.3 | 2.5 | 3.45 | 0.189 | 86.8 | Reaction slower than with pure C |
| E 11 | 335 | Gas absorpt. C saturated with $Cl_2$ | 30.4 [2] | 25 | 8.1 | 58.6 | 1.3 | 3.1 | 0.211 | 95.3 | Practically no reaction ∨ 335° |
| E 12 | 285 | Bone coal | 20 | 25 | 8.3 | 62.0 | 1.5 | 3.8 | 0.168 | 78.4 | Reaction slow [3] |
| E 13 | 335 | Wood charcoal | 20 | 25 | 2.5 | 27.0 | 0.6 | 10.35 | 0.123 | 83.2 | Reaction very slow |

[1] Without recycling of $C_2H_4Cl_2$.
[2] Original amount of C 25 g.
[3] About 1/2 or 1/3 of the reaction velocity observed for gas adsorption carbon (Merck).
[4] Assuming that all the substance in T is unreacted $C_2H_4Cl_2$. A destillation of the united residues from the experiments with gas adsorption carbon (Merck) gave a yield of about 95 % of substance with the boiling point of $C_2H_4Cl_2$, the rest being a viscous oil.



Table 10. *Further quantitative experiments with $C_2H_4Cl_2$ and gas adsorption carbon* (Merck) *as catalyst.*

Apparatus mainly according to fig. 2. Carbon always 25.0 g.

| Experiment No | T5 | T6 | T7 | T8 [10] | T9 [10] |
|---|---|---|---|---|---|
| React.temp., °C | 302—315 | 302—325 | 298—316 | 254—262 | 253—263 |
| » time, min. | 257 | 243 | 285 | 24 | 30 |
| Catalyst, added to carbon | — | — | $Br_2$, 1 g [15] | — $BaCl_2$, 12.4 g [14] | |
| $C_2H_4Cl_2$, g added | 101.1 | 101.0 | 99.6 | 26.2 | 28.3 |
| » unreacted after exp. in T | 5.4 | 5.0 | 13.9 | 1.4 | 2.0 |
| » total | 11.75 [3] | 11.8 [2] | 20.8 [8] | 22.5 [12] | 24.1 [13] |
| » net disappeared, g | 89.35 | 89.2 | 78.8 | 3.7 | 4.2 |
| » » moles | 0.903 | 0.901 | 0.796 | 0.037 | 0.042 |
| Yield of HCl, moles | 0.897 | 0.895 | 0.784 | 0.014 | 0.022 |
| » » » mole % of theoret. | 99.3 | 99.6 | 98.5 | 5.4 [1] | 8.6 [11] |
| Yield of vinyl chloride, total, g | 47.28 [1] | 49.7 [5] | 43.6 [7] | 0.9 [9] | 1.1 [9] |
| » » moles | 0.756 | 0.795 | 0.698 | 0.014 | 0.018 |
| » » » mole % | 83.7 | 88.2 | 87.7 | 5.4 [11] | 6.3 [11] |
| Increase of weight of catalyst, g | 1.6 | 1.0 [4] | — | —0.4 [6] | — |

[1] 47 g in V and 0.28 g bound in gas bromination products.
[2] 6.35 g $C_2H_4Cl_2$ was found in the vessel V. It was separated from $C_2H_3Cl$ by distillation.
[3] 1.9 g $C_2H_4Cl_2$(B.P. 84°) were extracted with ether from the gas scrubber flasks (HCl-flasks, WB) and 4.9 g $C_2H_4Cl_2$ were separated by distillation from the vinyl chloride in vessel V.
[4] A parallel experiment showed that 25 g of the carbon used lost 1.7 g in weight at heating in a glass tube at 300° with both ends open. Thus, the true amount of organic matter deposited in the catalyst during the experiment can be calculated to 1 + 1.7 = 2.7 g.
[5] 48.7 g in V, 0.6 g in the HCl flasks WB, 0.2 g in the $CaCl_2$-tube Ca and 0.19 g absorbed in the bromine.
[6] Weight of catalyst after exp. 24.6 g. Thus (note 4) the true amount of organic matter deposited can be calculated = —0.4 + 1.7 = 1.3 g.
[7] 42.6 g in V, assumed by deduction from results at T6 that 1.0 g of $C_2H_3Cl$ was absorbed in flasks WB and in Ca.
[8] 4.9 g $C_2H_4Cl_2$ in V; assumed by deduction from results of T6 that flasks WB contained 2.0 g of $C_2H_4Cl_2$.
[9] In V.
[10] Without recirculation of $C_2H_4Cl_2$.
[11] Calculated on the amount of $C_2H_4Cl_2$, which had passed the catalyst tube.
[12] 1.4 g in T + 21.1 g of $C_2H_4Cl_2$ which had passed the catalyst tube.
[13] 2.0 g in T + 22.1 g of $C_2H_4Cl_2$, which had passed the catalyst tube.
[14] The 25 g of carbon were impregnated with 14.6 g of $BaCl_2.2H_2O$ dissolved in 50 ml $H_2O$.
[15] Dissolved in the $C_2H_4Cl_2$ before experiment.

was impregnated with phosphotungstic acid and barium chloride respectively, whereas bromine was present at the experiment T 7. The experiments related in the tables 10 and 11 have been carried out by Dipl.Eng. Kerttu Turunen.

The experiments with zinc salts on silica gel (table 8) showed that the reaction, though fast to begin with, slows down very soon. As reaction products it gives an amount of HCl, which (exp. D1 and D7) is about one mole per mole of dichloroethane reacted, but practically no vinyl chloride. In the single case (exp. D 3, note 6) where the amount of gases formed was determined, these (unsaturated, $CO_2$, CO and $CH_4$) together were formed to the amount of about 1,9 millimoles only, whereas the amount of HCl developed was about 44 millimoles.

The bleaching earth Terrana (»Terrana extra») also split off hydrogen chloride very effectively, to the amount of about one mole per mole of reacted dichloroethane, in one case (exp. D 9) even somewhat more, but also did not form any vinyl chloride to speak of (exp. D 2). Unlike the zinc salts, Terrana formed considerable amounts of unsaturated compounds, in a case nearer investigated (exp. D 4, note 7, table 8) about 48 millimoles where 214 millimoles of HCl vere formed. Acetylene could not be detected (table 8, note 12). As other substances than ethylene were difficult to think of here, a new experiment was made in order to test on ethylene with 25.82 g = 261 millimoles of dichloroethane and 33.35 g of Terrana catalyst at 310°. This test, and also the later experiments with Terrana as catalyst, were made by Mr Viljo Veikko Kukkonen. No cold trap was used. The gases formed, in all 2810 ml at 20°C, were collected in two gasometer flasks. 360 millimoles of HCl were titrated in the gas scrubbing flasks, thus considerably more than one mole for each mole of dichloroethane. No chlorine could be detected in the flasks by the aid of potassium iodide and starch solution.

The main part (2085 ml) of the gas in the gasometer flasks was slowly led through scrubbing flasks containing 10 g of bromine, at room temperature for 9 hours. A heavy oil was formed in the flasks. It was separated off, washed twice with diluted sodium hydroxide solution and thereafter with water, dried with calcium chloride and distilled without column with the following result: B.P.80—130° 1.30 g, 130—140° 5.00 g, 138—144° 2.62 g, residue 0.93 g. The fraction with B.P. 130—140° showed d = 2.151 and $n_D^{20} = 1.5424$. The fractions with B.P. 130—140° and 138—144° were united, washed anew with sodium hydroxide solution and water, dried and distilled. Practically all the substance distilled between 131 and 140° and showed B.P. by the Emich micro method of 131°. A comparison with the constants of i.a. ethylene bromide, acetylene tetrabromide and the addition product $C_2H_3ClBr_2$ of vinyl



chloride and bromine shows that the bromination product investigated must mainly consist of ethylene bromide. Supposing that the whole oil isolated (9.85 g) had consisted of ethylene bromide, that would mean that the gaseous reaction products had contained at least 70.6 millimoles of ethylene.

*Experiments with active carbon as catalyst.*

The experiments with active carbon as catalyst for the fission of dichloroethane (tables 8—11) gave yields of up to 99.5 mole % of hydrogen chloride and up to 88.2 mole % of vinyl chloride. The yield of vinyl chloride is thus very substantial with active carbon, contrary to what is the case at the reaction with zinc salt or Terrana catalyst. There is, however, a difference of about 10 % between the yields of vinyl and hydrogen chloride. At the experiments T5 and T6 (Table 10) the possible cause of this difference was attentively studied, especially in order to find out to what extent ethylene is formed with active carbon catalyst.

Exp. T5. The apparatus was mainly the same as that pictured in fig. 2. The straight reflux condenser R with its inner cooling tube was, however, exchanged for a condenser with spiral formed inner tube, and the calcium chloride tube Ca was considerably bigger than the formerly used one. The gases which did not condense in the cold trap (vessel V), were conducted through two scrubbing flasks containing bromine and water, thereafter through a flask containing sodium hydroxide solution and at last collected in a gasometer flask.

The vinyl chloride collected in the vessel V was identified by vaporization at room temperature and leading the vapors through scrubbing flasks containing bromine and water at room temperature. The bromine was consumed and a considerable rise of the temperature was observed. The reaction product was an oil, which was washed with water and distilled. It showed B.P. 163° and d 20/4 2.2785. It obviously consisted mainly of the addition product of vinyl chloride and bromine $CH_2Br.CHClBr$, which has B.P. 162.5° and d 2.268 [107].

The reaction product with bromine of the gas, which did not condense in the cold trap, was an oil, which after washing with water, drying and fast distillation without a column gave a fraction of 1.89 g boiling below 143° (probably mainly $C_2H_3Br_2$) and showing the saponification number 295 mg KOH/g, and another fraction of 1.01 g boiling at 152—166° (probably mainly $C_2HCl_4Br_2$).

Exp. T6. The experiment was conducted in the same apparatus as T5. The reaction product with bromine of the gas which did not condense in the cold trap was an oil, which after washing

with water weighed 1.95 g. It was distilled twice using a Vigreux column. The main fraction comprised about 2/3 of the whole oil and showed B.P. (corr.) 127—134° and $n_D$ 1.535. Thus this fraction obviously consisted mainly of ethylene bromide, and the gas had contained ethylene. Assuming that the higher boiling fraction consisted of $C_2H_3ClBr_2$ and that the proportions of the $C_2H_4Br_2$ and $C_2H_3ClBr_2$ fractions were the same as in exp. 5, the amount of ethylene in the gas can be calculated to about 0.19 g, and that of uncondensed vinyl chloride reacted to $C_2H_3ClBr_2$ also to about 0.19 g.

Gas not condensed, which had not reacted with bromine, was collected into an amount of 2750 ml. It was analyzed in an Orsath apparatus and was found to contain 94.0 % $N_2$, 1.3 % $O_2$, unsaturated compounds 0.2 %, $CO_2$ 0.2 %, CO 3.9 %, and $H_2$ 0.4 %. No methane could be detected. The total amount of carbon dioxide developed at the experiment can be greater, as this gas could have been absorbed in the sodium hydroxide solution (see preceeding page) which it had passed on its way to the gasometer flask.

Table 11 gives a substance bilance for the exp. 6, where also the amounts of dichloroethane and vinyl chloride respectively found in the different parts of the apparatus are observed. On

**Table 11.** *Substance bilance at experiment T6 with active carbon as catalyst for the fission of dichloroethylene.*

| | g | moles | mole % |
|---|---|---|---|
| Input of $C_2H_4Cl_2$ | 101.0 | | |
| Unreacted $C_2H_4Cl_2$ after reaction: | | | |
| in flask T | 5.0 | | |
| in cold trap vessel V | 4.9 | | |
| in gas scrubbing(HCl-)flasks WB | 1.9 | | |
| Total | 11.8 | | |
| $C_2H_4Cl_2$ consumed | 89.2 | 0.901 | 100 |
| Vinyl chloride found: | | | |
| in cold trap vessel V | 48.70 | 0.779 | 86.5 |
| in scrubbing flasks WB | 0.60 | 0.010 | 1.1 |
| in $CaCl_2$ tube Ca | 0.20 | 0.003 | 0.3 |
| absorbed in $Br_2$ | 0.19 | 0.003 | 0.3 |
| Total | 49.69 | 0.795 | 88.2 |
| Gases formed: | | | |
| ethylene, absorbed in $Br_2$ | 0.19 | 0.007 | 0.8 |
| CO | | 0.004 | 0.4 |
| $CO_2$, at least | | 0.004 | 0.4 |
| Substance absorbed of catalyst, calculated as $C_2H_3Cl$ | 2.7 | 0.043 | 4.8 |
| Substance unaccounted for | | 0.052 | 5.8 |
| Sum | | 0.901 | 100.0 |
| Yield of HCl | | 0.897 | 99.6 |



this connection also a control distillation with a Vigreux column of 49.35 g of the used 1,2-dichloroethane (kindly delivered from Kymmene AB, originating from O.Y. Ahlström AB, Warkaus) can be reported: distillate B.P. (corr.) 82.7—84.2° 96.1 %, residue in the flask 2.2 %, loss 1.7 %. No effervescence of gas bubbles at the beginning of the destillation (ethylene) could be detected.

It can be deduced that the amount of acetylene formed at exp. 6 cannot be great, as it would appear in the gases not condensed in the cold trap together with ethylene and a small amount of vinyl chloride, and would react with bromine forming acetylene tetrabromide. Even if the bromination product distilling over 134° had consisted entirely of acetylene tetrabromide, the amount of acetylene would have been only 0.2 mole %.

The loss ascribed in table 11 to the weight increase of catalyst, 4.8 mole % calculated as vinyl, chloride is far bigger. Almost two thirds of this value, however, is calculated on the basis of a blank drying experiment (table 10, note 4), and the increase of the weight of the catalyst according to the experiments E1—E9 in table 9 is by no means proportional to the amount of dichloroethane used, but generally smaller in mole % the greater the amounts of dichloroethane passed through the catalyst tube, being for instance 2.9 mole % at exp. E7 and 2.3 mole % at exp. E6.

The deposition of substance on the catalyst can be feared to cause fouling of the same. Doroswany and Pai [84] have observed rather bad fouling of active carbon catalysts for dichloroethane fission at 435° and conclude, that active carbons do not appear to be suitable catalysts for the production of vinyl chloride from dichloroethane. At the present work, however, no fouling, but rather a certain activation could be observed at 310° at compared with the results of the experiments E6 and E7 (table 9), where the same 25 g of active carbon were used successively for tree portions of 125 g of dichloroethane. When, at exp. E9, the same catalyst was further used at 285°, a slight fouling could be observed in the once through yield in comparison with the experiment E8, where fresh catalyst was used.

The influence of temperature is clearly shown by a comparison of exp. T8 at about 255° (table 10) with E8 and E9 at 285° (compare also E2 with E3) and exp. 112—114, table 3, the once through yield being about six times greater at the higher temperature. It seems questionable whether there is any advantage in using 310° instead of 285° (compare E7 with E5, E4 with E3).

Impregnation of the carbon with phosphotungstic acid seems to deteriorate the catalyst (compare E10 with E3 and E5 and also exp. 54, table 4), and likewise the saturation of the carbon with chlorine (exp. E11). Addition of some bromine to the dichloroethane (exp. T7, table 10) does not seem to influence

the reaction, but impregnation of the carbon with about 50 % of its weight of barium chloride seems to increase the once through yield of vinyl chloride slightly but also the difference between the yields of vinyl and hydrogen chloride (compare T8 with T9). Bone coal and wood charcoal react considerably slower than gas adsoprtion coal (Merck) (exp. E12 and E13).

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# The Radioisotope Laboratory of the Finnish Pulp and Paper Research Institute*

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This laboratory, which has recently been taken into use, comprises one preparation room, or »cell», one counting room, and one dark room for autoradiography. So far only microcurie amounts of radioactivity have been handled in the experiments, and the total quantity kept in store does not generally exceed 10 millicuries. The facilities provided in the cell in order to avoid danger to health and contamination can be considered as more than sufficient. It should, however, be mentioned that much of the work carried out involves the use of hard gamma emitters.

The walls and ceiling of the cell are coated with hardboard plates, which are screwed on a wooden lattice-work. The plates are painted with a plastic-base »stripping paint», which can easily be removed and renewed in the event of contamination. Should a plate become seriously contaminated, it can be replaced by a new one. The floor is coated with vinyl sheets, which are also exchangeable. »Telon» sheets are employed as cover for the working surfaces of the benches. In order to avoid unnecessary accumulation of dust, the electricity supply, the water and gas lines, and so on, are located in the lattice-work.

The box for storage of the radioactive material is made from iron plate. Both box and door are covered by a 20-cm thick layer of magnetite concrete. The hinges of the rather heavy door are provided with ball bearings.

The »magnetite concrete» is made from a mixture of two parts (by volume) of fine-grade magnetite ore and one part (by volume) of cement.

Lead bricks and blocks made from magnetite concrete are used as mountable shields against gamma rays. The magnetite

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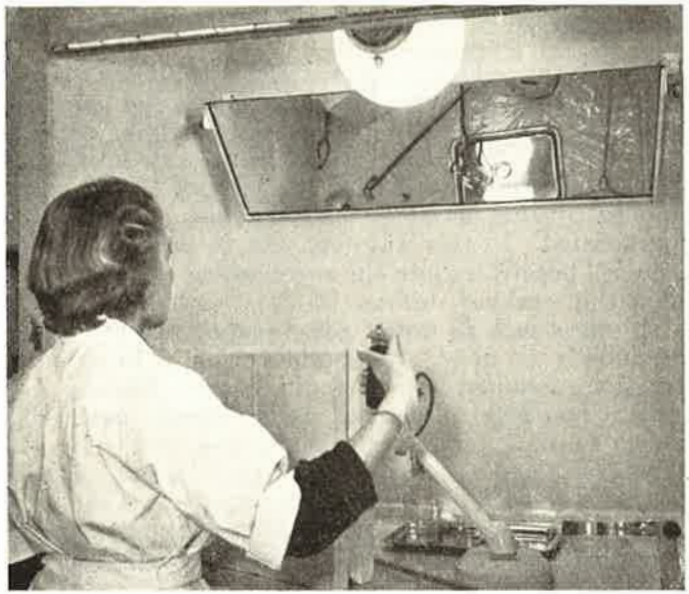

Fig. 1. Hood.

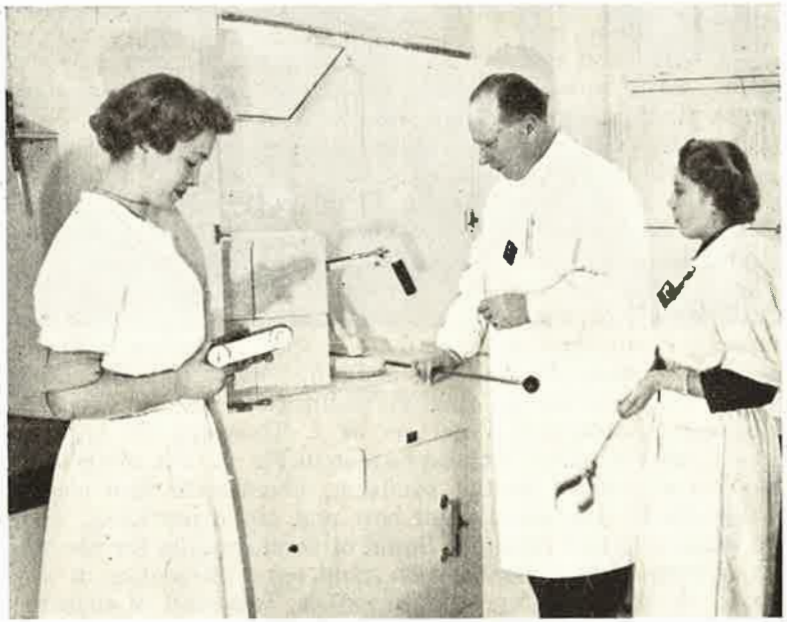

Fig. 2. Some Remote Handling Tools.

blocks are designed in such a way as to leave no direct paths for radiation at the joints.

The hood in the cell (fig. 1) is provided with forced ventilation. The flow of air is about one-half cubic metre per second. The exhausted air is filtered through three dust filters arranged in series. In the outer wall of the cell, there is an air inlet, provided with a fan and means for filtering and heating. The quantity of air supplied by this device is somewhat less than the amount being exhausted. In this way, one can be certain that no air from the cell penetrates into the surrounding rooms. The hood, including the working surface, is also painted with stripping paint. Supplies such as water, gas, vacuum, and electricity are located outside the hood. If a gamma shield is being used, the operations are followed by means of a mirror. Various tools for the remote handling of sources are frequently used (Fig. 2), such as for example tongs, pipetting devices, and so on. In the hood opening there are hinged 1/2″ thick sheets of perspex, which when turned down cover about one-half of the opening. They are used as beta shields, if necessary in combination with mountable shields made of similar material. If dust-forming substances are being dealt with, dry boxes are used. In practical work in general, the working surface of the hood is covered by means of a polyethylene foil, and the operations are carried out on stainless steel trays which are covered with polyethylene and absorbent tissue.

The main sink is provided with a three-way valve. By this means, waste water which has too high a level of radioactivity can be drained into separate vessels.

The radioactive waste is concentrated as much as possible, as for example by means of mixed-bed ion exchangers. Short-lived materials are then stored until the activity has decayed sufficiently, and long-lived waste is buried in accordance with the regulations made by the public authorities.

At present, the counting equipment consists of a relatively small number of units, which were chosen so as to meet the more important requirements. There are in use two scalers, a Tracerlab »Superscaler» (Fig. 3) and a Philips »Elektronisches Zählgerät». The former is used in connection with a Tracerlab »Gamma Scintillation Detector» or a Tracerlab »Windowless Flow Counter», which can also be seen in Fig. 3. The scintillation detector is used for gamma counting, whereas the flow counter is suitable for the counting of beta and alpha particles. There are various Geiger tubes for liquid or solid samples for use with both scalers. For contamination monitoring (inspection of work areas, clothing, wastes, and so on) a Tracerlab »Laboratory Monitor» is employed, this being provided with a ratemeter and a Geiger tube sensitive to both beta and gamma radiation. The



determination of the radiation dose rate, in roentgens or milliroentgens per hour, is carried out by means of a battery-operated Frieseke & Hoepfner »Radiameter». In order to obtain information as to the amount of radiation to which personnel have been exposed during a certain period of time, film badges and pen dosimeters are worn by the workers concerned.

Amongst items of equipment which will probably be procured in addition, apparatus for pulse height analysis may be mentioned.

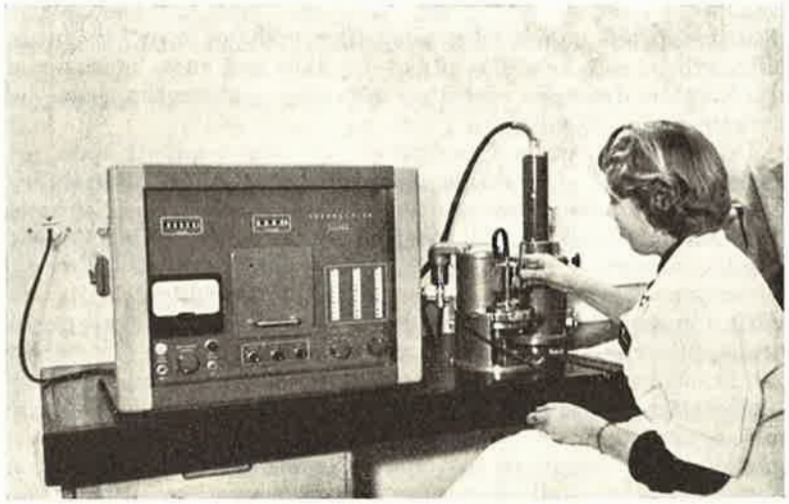

Fig. 3. A Scaler, a Windowless Flow Counter, and a Gamma Scintillatio Detector.

# Vilka kemiska tidskrifter citeras oftast?

*(Which Journals Are Most Cited Frequently by Chemists?)*

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Under årens lopp har flere statistiska studier publicerats rörande den frekvens med vilken olika kemiska tidskrifter citeras i fackpressen. Dylika statistiska undersökningar motiveras i allmänhet med två argument. Dels kan man på basen av ett dylikt material avgöra vilka tidskrifter, som det är av vikt att ha tillgängliga i ett kemiskt bibliotek. Dels kan man också ur en dylik studie dra vissa slutsatser rörande språkkunskapernas betydelse för en kemist.

De flesta tidigare undersökningar har berört amerikanska tidskrifter, främst *Journal of the American Chemical Society* [4,7,1] och *Industrial and Engineering Chemistry* [6,1]. Ett par arbeten har också inskränkt sig till speciella grenar av kemin (agrikulturkemi [3], analytisk och organisk kemi [2]). *Recueil des Travaux Chimiques des Pays-Bas* är den enda europeiska tidskrift, som varit föremål för en studie i detta avseende [5]. Det kan därför anses motiverat att utföra en liknande undersökning beträffande någon annan europeisk tidskrift. Ett lämpligt objekt erbjuder utan tvivel *Acta Chemica Scandinavica*. Speciellt ur nordisk synpunkt är det av intresse att lära känna förhållandena i vår ledande tidskrift på området. Dessutom får väl *Acta Chem. Scand.*, vars innehåll omspänner alla grenar av kemin, betraktas som en typisk representant för den europeiska tidskriftsfloran på kemins område.

I föreliggande studie har jag som undersökningsobjekt valt årgångarna 1, 5 och 10 av *Acta Chem. Scand.* och materialet sträcker sig sålunda över tioårsperioden 1947—1956. I dessa tre årgångar är det totala antalet citeringar 6 430, fördelade på mer än 300 olika tidskrifter. Ett mycket stort antal av dessa tidskrifter uppträder blott ett fåtal gånger. Å andra sidan finns det ett fåtal tidskrifter, som citeras synnerligen ofta. Sålunda hänför sig drygt hälften av alla referenser till blott nio olika tidskrifter och om de allmännast citerade tidskrifternas antal ökas till 21 täckes tvåtredjedelar av referenserna. De tidskrifter, som i



de tre berörda årgångarna av *Acta Chem. Scand.* citerats sammanlagt 30 gånger eller mera, finns upptagna i Tabell 1.

Det mest iögonenfallande faktum, som kan utläsas ur tabellen, är att de flesta referenserna (15 %) hänför sig till andra uppsatser i *Acta Chem. Scand.* I detta hänseende förhåller sig tidskriften normalt. Ur samtliga tidigare undersökningar har det nämligen framgått, att en tidskrift oftast citerar sig själv. Orsaken härtill är väl delvis den, att författare ofta publicerar sina resultat i en och samma tidskrift och därvid i hög grad citerar egna tidigare arbeten. Frekvensen av »självciteringar» i *Acta Chem. Scand.* överensstämmer också kvantitativt rätt väl med tidigare publicerade siffror för andra tidskrifter. Dessa varierar för det mesta mellan 15 och 20 %. En extremt hög frekvens av självciteringar förekommer i *J. Am. Chem. Soc.* för år 1955, där *Barrett & Barrett* [1] beräknat dem till i det närmaste 40 %.

Om man går vidare i förteckningen i Tabell 1 följer på andra till femte plats *Journal of the American Chemical Society, Journal of Biological Chemistry, Berichte* och *Journal of the Chemical Society* i nämnd ordning. Dessa fyra tidskrifter är även de oftast citerade i *J. Am. Chem. Soc.* för år 1955 ehuru den inbördes ordningsföljden är en annan. Tre av dessa tidskrifter *(J. Am. Chem. Soc., J. Chem. Soc.* och *Ber.)* toppar också förteckningarna för *J. Am. Chem. Soc.* 1926 [4] och 1933 [7] samt *Rec. trav. chim.* 1937—39 [5]. Det är anmärkningsvärt att *J. Biol. Chem.* under det senaste årtiondet synbarligen ryckt fram till en rangställning likvärd med dessa tre klassiska tidskrifter. Orsaken härtill står väl att finna i biokemins kraftiga expansion under den senaste tiden. Denna omständighet framgår även av att drygt 17 % av samtliga referenser i de undersökta årgångarna av *Acta Chem. Scand.* hänför sig till rent biokemiska tidskrifter, av vilka sju olika citerats mer än 30 gånger.

Vid en jämförelse av de i Tabell 1 uppräknade tidskrifterna med dem i tidigare publicerade förteckningar, finner man att 15 av de 25 första tidskrifterna i tabellen återfinns bland de 25 oftast citerade tidskrifterna i *J. Am. Chem. Soc.* 1955. För årgång 1933 av samma tidskrift och årgångarna 1937—39 av *Rec. trav. chim.* är motsvarande siffra 13. Härav framgår det tydligt att det är ett relativt begränsat antal tidskrifter, som står för en överväldigande del av citeringarna i den kemiska litteraturen. I stort sett är förhållandena i *Acta Chem. Scand., J. Am. Chem. Soc.* och *Rec. trav. chim.* rätt likartade. Den största skillnaden står självfallet att finna i ett markant nordiskt inslag i *Acta Chem. Scand.* T.ex. i *J. Am. Chem. Soc.* 1955 förekommer ej någon nordisk tidskrift bland de 25 oftast citerade. I *Acta Chem. Scand.* förekommer däremot bland de 30 första 6 nordiska tidskrifter. Näst *Acta Chem. Scand.* själv kommer *Arkiv för Kemi* på sjunde plats, *Svensk Kemisk Tidskrift* på elfte och längre ned

*Tabell 1.* De oftast citerade tidskrifterna i *Acta Chemica Scandinavica* 1947, 1951 och 1956.

| Tidskrift | Antal citeringar | | | Totalt | % |
|---|---|---|---|---|---|
| | Vol. 1 1947 | Vol. 5 1951 | Vol. 10 1956 | | |
| 1. Acta Chem. Scand. | 31 | 296 | 629 | 956 | 14,9 |
| 2. J. Am. Chem. Soc. | 77 | 137 | 373 | 587 | 9,1 |
| 3. J. Biol. Chem. | 54 | 243 | 219 | 516 | 8,0 |
| 4. Ber. | 93 | 108 | 78 | 279 | 4,3 |
| 5. J. Chem. Soc. | 37 | 83 | 155 | 275 | 4,3 |
| 6. Biochem. J. | 44 | 99 | 83 | 226 | 3,5 |
| 7. Arkiv Kemi | 35 | 45 | 87 | 167 | 2,6 |
| 8. Ann. | 38 | 40 | 60 | 138 | 2,2 |
| 9. Nature | 19 | 54 | 57 | 130 | 2,0 |
| 10. Z. phys. Chem. | 44 | 32 | 40 | 116 | 1,8 |
| 11. Svensk Kem. Tidskr. | 39 | 28 | 36 | 103 | 1,6 |
| 12. Helv. chim. Acta | 18 | 36 | 49 | 103 | 1,6 |
| 13. Z. anorg. allg. Chem. | 28 | 15 | 60 | 103 | 1,6 |
| 14. Biochem. Z. | 43 | 34 | 21 | 98 | 1,5 |
| 15. Arch. Biochem. Biophys. | 11 | 39 | 29 | 79 | 1,2 |
| 16. Hoppe-Seyler's Z. physiol. Chem. | 37 | 24 | 11 | 72 | 1,1 |
| 17. Anal. Chem. | 7 | 19 | 46 | 72 | 1,1 |
| 18. Biochim. et Biophys. Acta | 1 | 16 | 53 | 70 | 1,1 |
| 19. Kolloid-Z. | 22 | 12 | 29 | 63 | 1,0 |
| 20. Proc. Roy. Soc. (London) | 16 | 13 | 26 | 55 | 0,9 |
| 21. Trans. Faraday Soc. | 10 | 7 | 38 | 55 | 0,9 |
| 22. J. prakt. Chem. | 17 | 11 | 20 | 48 | 0,7 |
| 23. Rec. trav. chim. | 13 | 14 | 20 | 47 | 0,7 |
| 24. Suomen Kemistilehti | 21 | 7 | 17 | 45 | 0,7 |
| 25. J. Phys. Chem. | 7 | 9 | 28 | 44 | 0,7 |
| 26. Bull. soc. chim. France | 6 | 12 | 24 | 42 | 0,7 |
| 27. Z. Elektrochem. | 12 | 4 | 25 | 41 | 0,6 |
| 28. Science | 6 | 19 | 16 | 41 | 0,6 |
| 29. Acta Physiol. Scand. | 14 | 13 | 13 | 40 | 0,6 |
| 30. Tidsskr. Kjemi, Bergv. Met. | 34 | 0 | 5 | 39 | 0,6 |
| 31. J. Chem. Phys. | 4 | 6 | 27 | 37 | 0,6 |
| 32. Compt.rend. | 7 | 14 | 13 | 34 | 0,5 |
| 33. Z. Naturforsch. | 0 | 5 | 25 | 30 | 0,5 |
| Övriga | 369 | 555 | 755 | 1 679 | 26,2 |
| Summa | 1 214 | 2 049 | 3 167 | 6 430 | |

på rangskalan ytterligare *Suomen Kemistilehti*, *Acta physiologica Scandinavica* och *Tidsskrift för Kjemi, Bergvesen og Metallurgi*.

Ur Tabell 1 kan man även se i vilken mån citeringsfrekvensen för de olika tidskrifterna fluktuerar under loppet av den undersökta tioårsperioden. I en ung tidskrift som *Acta Chem. Scand.* är det naturligt att frekvensen av självciteringar ökar med åren. Parallellt härmed minskar antalet referenser till de flesta av de övriga nordiska tidskrifterna, som självfallet fått finna sig i en



minskad internationell betydelse sedan *Acta Chem. Scand.* börjat utges. Ett undantag utgör *Arkiv för Kemi*, som bevarat sin ställning oförändrad.

Det är anmärkningsvärt att referenserna till *J. Am. Chem. Soc.* starkt ökat under den senaste tiden. Sedan den första årgången av *Acta Chem. Scand.* (1947) har den procentuella andelen av referenser till *J. Am. Chem. Soc.* i det närmaste fördubblats och uppgick 1956 till 11,9 %. Denna ökning har främst skett på bekostnad av de tyska tidskrifterna, av vilka det egentligen endast är *Liebieg's Annalen*, som lyckats någorlunda bevara sin ställning. *Berichte* har gått katastrofalt tillbaka. Från att år 1947 ha varit den oftast citerade tidskriften i *Acta Chem. Scand.*, sjönk dess ställning 1951 till fjärde och 1956 till sjunde plats. Ännu större är tillbakagången beträffande de båda tyska biokemiska tidskrifterna (*Biochemische Zeitschrift* och *Hoppe-Seyler's Zeitschrift für physiologische Chemie*).

De tidskrifter, som visar den största konstansen beträffande citeringsfrekvensen under den ifrågavarande perioden är i nämnd ordning: *Helvetica Chimica Acta*, *Arkiv för Kemi*, *Bulletin de la Société de Chimie de France*, *Nature*, *Journal of the Chemical Society* och *Liebig's Annalen*.

För att ge en viss uppfattning om den geografiska fördelningen hos de citerade tidskrifterna har Tabell 2 sammanställts. Denna upptar antalet citerade tidskrifter och antalet citeringar i de tre årgångarna av *Acta Chem. Scand.* med hänsyn till utgivningsland. Dessutom anges för varje land den procentuella andelen i det totala antalet citeringar.

Om man betraktar de tre undersökta årgångarna av *Acta Chem. Scand.* som en helhet, leder de i U.S.A. utgivna tidskrifterna klart med 29,7 % av samtliga citat. På andra plats kommer Tyskland med 19,9 % och därefter de samnordiska tidskrifterna (16,5 %) och de brittiska (inkl. dominions, 14,6 %). Dessa fyra grupper svarar för drygt 80 % av samtliga citat. Ordningsföljden mellan dessa fyra utgivarländer har avsevärt förändrats under den ifrågavarande tioårsperioden. År 1947 intogs främsta platsen med god marginal av Tyskland (36 %). Antalet tyska tidskrifter som citerats har hållit sig praktiskt taget oförändrat, men antalet citeringar har nedgått, så att Tyskland år 1951 kom på andra och 1956 på tredje plats. Tysklands tidigare dominerande ställning har intagits av U.S.A. Antalet referenser till amerikanska tidskrifter steg från 20 % år 1947 till 32 % år 1951 och har sedan hållit sig konstant. Antalet citerade tidskrifter har under tioårsperioden nästan fördubblats. Referenserna till brittiska tidskrifter har däremot hållit sig mycket konstant. Detsamma gäller för övrigt också de flesta andra europeiska länder (Nederländerna, Schweiz, Frankrike). De samnordiska tidskrifterna har gått starkt framåt vad antalet citat beträffar,

*Tabell 2.* Antalet citeringar och citerade tidskrifter i *Acta Chemica Scandinavica* 1947, 1951 och 1956 sammanställda enligt utgivningsland.

| Utgivningsland | Antal citeringar och tidskrifter | | | | | | Totala antalet citeringar | % |
|---|---|---|---|---|---|---|---|---|
| | Vol. 1 1947 | | Vol. 5 1951 | | Vol. 10 1956 | | | |
| 1. U.S.A. | 239 | (40) | 661 | (72) | 1 010 | (77) | 1 910 | 29,7 |
| 2. Tyskland | 433 | (47) | 386 | (52) | 477 | (46) | 1 278 | 19,9 |
| 3. Samnordiska | 63 | (10) | 334 | (9) | 662 | (8) | 1 059 | 16,5 |
| 4. Storbritannien * | 167 | (28) | 302 | (26) | 470 | (36) | 939 | 14,6 |
| 5. Sverige | 92 | (13) | 117 | (20) | 147 | (12) | 356 | 5,5 |
| 6. Frankrike | 39 | (12) | 48 | (13) | 64 | (14) | 151 | 2,4 |
| 7. Nederländerna | 25 | (7) | 39 | (8) | 86 | (11) | 150 | 2,3 |
| 8. Schweiz | 26 | (4) | 53 | (9) | 53 | (3) | 132 | 2,1 |
| 9. Finland | 31 | (5) | 24 | (7) | 44 | (7) | 99 | 1,5 |
| 10. Danmark | 22 | (4) | 14 | (5) | 31 | (8) | 67 | 1,0 |
| 11. Japan | 3 | (3) | 18 | (9) | 46 | (16) | 67 | 1,0 |
| 12. Norge | 52 | (5) | 3 | (2) | 11 | (5) | 66 | 1,0 |
| 13. Sovjet | 7 | (4) | 17 | (10) | 21 | (11) | 45 | 0,7 |
| 14. Italien | 4 | (4) | 10 | (4) | 14 | (8) | 28 | 0,4 |
| 15. Österrike | 1 | (1) | 8 | (1) | 8 | (2) | 17 | 0,3 |
| 16. Indien | 2 | (1) | 7 | (3) | 7 | (3) | 16 | 0,3 |
| 17. Belgien | 4 | (3) | 16 | (6) | 3 | (2) | 13 | 0,2 |
| 18. Tjeckoslovakien | 2 | (2) | 2 | (2) | 7 | (3) | 11 | 0,2 |
| 19. Spanien | 0 | (0) | 4 | (3) | 3 | (2) | 7 | 0,1 |
| 20. Argentina<br>21. Kina<br>22. Polen<br>23. Indonesien<br>24. Rumänien<br>25. Turkiet | 2 citeringar var<br>1 citering var | | | | | | 9 | 0,2 |

trots att de citerade tidskrifternas antal ej har ökat. Ökningen har till övervägande del skett på bekostnad av de nordiska ländernas egna tidskrifter. Detta framgår av att det totala antalet referenser till nordiska tidskrifter under de tre ifrågavarande åren ökat i mycket mindre grad (21, 24, 28 %). De norska tidskrifterna har stått sig sämst i konkurrensen, medan de svenska lyckats bibehålla en relativt god position. Finland och Danmark intar en mellanställning.

Vidare kan det vara av intresse att notera, att Japan gått mycket starkt framåt både vad antal tidskrifter och citat beträffar. Referenserna till ryska tidskrifter har däremot hållit sig konstant. Beträffande de övriga utgivarländerna finns det knappast något av intresse att tillägga.

På basen av Tabell 2 kan man även få en antydan om den språkliga fördelningen av de citerade uppsatserna. Inslaget av

* Inklusive dominions.



flerspråkiga internationella tidskrifter har emellertid med åren blivit allt större och utgivarlandets språk behöver därför ej vara identiskt med tidskriftens huvudspråk. Även tidskrifternas namn kan leda till missuppfattningar i detta avseende, som t.ex. då en författare [7] drar slutsatsen att latin är huvudspråket i *Annales Academiae Scientiarum Fenniae.*

Tidskrifter publicerade i U.S.A. och Storbritannien kan utan vidare betraktas som engelskspråkiga. Dessa utgör tillsammans 44 %. De flerspråkiga internationella tidskrifternas andel är uppskattningsvis 20 %. Minst hälften av uppsatserna i dessa kan anses vara engelskspråkiga. Detta betyder att minst 54 % av de i *Acta Chem. Scand.* citerade uppsatserna är engelskspråkiga. De tyska uppsatsernas antal når knappast mer än till 25 %, de franska ej ens till 10 % och de slaviska språkens andel rör sig kring 1 %. Härav framgår det att den engelska dominansen är mycket framträdande, om också inte på långt när lika överväldigande som i en del amerikanska tidskrifter: *J. Am. Chem. Soc.* 1955 över 75 % och *Ind. Eng. Chem.* 1955 minst 87 %. Det tyska inslaget är å andra sidan också så pass stort, att kunskaper i tyska måste anses obetingat nödvändiga för en kemist, som vill följa med litteraturen på sitt område. Beträffande t.ex. franska och ryska tidskrifter är citeringsfrekvensen förvånansvärt låg. Det totala antalet referenser på dessa språk är trots allt rätt ansenligt och det är inte heller uteslutet att uppsatser på dessa och andra mindre kända språk förbigås på grund av bristande språkkunskaper. Alltför långtgående slutsatser kan man därför inte dra av en dylik undersökning. I likhet med vad fallet varit vid tidigare undersökningar av detta slag får man inskränka sig till att i största allmänhet poängtera språkkunskapernas betydelse för en kemist.

## Summary

The paper deals with a statistical study of the frequency of citations to various chemical journals in volumes 1, 5, and 10 of *Acta Chemica Scandinavica.* The total number of citations was 6 430 and more than 300 journals were cited. Next to the *Acta Chem. Scand.* itself, the ten most cited journals are, in order: *Journal of the American Chemical Society, Journal of Biological Chemistry, Berichte, Journal of the Chemical Society (London), Biochemical Journal, Arkiv för Kemi, Liebig's Annalen, Nature, Zeitschrift für physikalische Chemie,* and *Svensk Kemisk Tidskrift.* About 30 per cent of the articles cited are in journals issued in the United States and about 25 per cent of the citations concern Scandinavian journals. These are followed by Germany

(20 %), Great Britain (15 %) and France (2 %). The variations in the frequency with which different journals are cited is discussed.

## Litteratur


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# Förteckning över Finska Kemistsamfundets medlemmar den 31. 12. 1957

# Suomen Kemistiseuran jäsenluettelo 31. 12. 1957

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