

Investigation of Tritium Removal by means of
Organic Compounds. Catalytic Hydrogenation
(Tritiation) of Linoleic Acid

A. El-Sharnouby⁺, H. Weichselgartner

IPP 1/234

November 1984



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*Die nachstehende Arbeit wurde im Rahmen des Vertrages zwischen dem
Max-Planck-Institut für Plasmaphysik und der Europäischen Atomgemeinschaft über die
Zusammenarbeit auf dem Gebiete der Plasmaphysik durchgeführt.*

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Abstract

In the presence of noble-metal catalysts unsaturated fatty acids such as erucic acid and linoleic acid capture hydrogen (and tritium) quantitatively. The hydrogenation reaction of erucic acid has already been reported /1/.

The experimental results of the reaction of hydrogen (and tritium) with linoleic acid are now discussed in this paper. Obviously, the use of linoleic acid shows some advantages compared with erucic acid:

- the hydrogenation reaction is faster,
- linoleic acid is liquid, so that the choice of additional solvents is easier, and
- linoleic acid is a more or less cheap natural product, which is available from a series of seeds, so that the cost of a technical tritium removal plant is not increased by the basic chemical material.

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1. Introduction

We have already reported on catalytic hydrogenation of erucic acid /1/. These investigations of tritium removal by means of organic compounds have now been continued with linoleic acid as a follow-up to previous work /2/.

Linoleic acid is a natural, unsaturated fatty acid with the chemical formula $C_{17}H_{31}-COOH$.

In the carbon chain it has two double bonds, which gives it the structure formula $HC_3-(CH_2)_4-CH=CH-CH_2-CH=CH-(CH_2)_7-COOH$.

Chemically speaking, it is cis-9 cis-12-octadecadienoic acid.

Linoleic acid is derived on a large scale from vegetable oils /3/ such as coconut oil, palm oil, rapeseed oil, soya oil and sunflower-seed oil and is one of the unsaturated fats essential to human nourishment which are mainly consumed in the form of salad oil and margarine.

Should the investigation yield a technically applicable method, the abundance and cheapness of linoleic acid will be a distinct advantage.

As linoleic acid is liquid at room temperature, it was possible to investigate the influence of the solvent in more detail than in the case of erucic acid.

Meanwhile, test results with a semi-technical reaction column are also available.

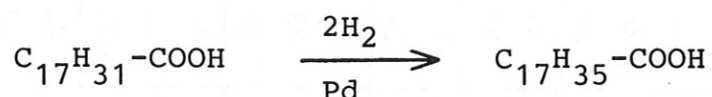
2. Experimental

The experimental implementation of catalytic hydrogenation has already been described in detail in a previous work /1/. First the catalyst, then the mixture of solvent and linoleic acid, and finally the hydrogen or tritium were put into gas mice (60 mm long and 50 mm in diam.) and shaken (50 r.p.m.). Samples were taken from the gas mice at defined time intervals to determine the hydrogen or tritium concentration. The hydrogen concentration was determined

by gas chromatography, and the tritium concentration by measuring the activity by means of liquid scintillation. The absorption of T by the solvent and catalyst was determined in dummy tests. For this purpose calibration standards of the J.T. Baker B.V. company [Instand $^3\text{H-O}$: glycerol tripalmitate- $9 \cdot 10^{-3}\text{H(n)}$] was used.

3. Results and discussion

Hydrogenation of linoleic acid is an irreversible process



yielding stearic acid, a compound which is solid at room temperature. The results were obtained under various test conditions. They are described in detail in the following and documented in Table 1 and Figs. 1 to 8. The hydrogen (tritium) concentration in relation to the total reaction volume is plotted in $\mu\text{l/ml}$ ($\mu\text{Ci/ml}$) as a function of the reaction time in minutes.

3.1 Influence of the linoleic acid concentration

First the influence of the linoleic acid concentration on the hydrogenation rate in the presence of diethylene glycol dimethyl ether $\text{C}_8\text{H}_{18}\text{O}_3$ solvent was investigated in Exp. 1, 2 and 3 and represented in Fig. 1. The linoleic acid concentration was raised from 1 g in Exp. 1 to 2 g and 4 g in Exp. 2 and 3, respectively. From Fig. 1 it is seen that the reaction time decreases as the linoleic acid concentration increases.

The hydrogenation times are 43, 33 and 30 min, respectively, i.e. when the quantity of linoleic acid used is doubled and quadrupled, the reaction time is shortened by 23 and 30 %, respectively. Comparison of Exp. 2 and 3 shows, however, a time reduction of just 9 %.

Experiments 4 and 5 show the influence of the linoleic acid concentration on the hydrogenation rate without solvent. The linoleic acid was increased from 10 g in Exp. 4 to five times that amount in Exp. 5. Large quantities of linoleic acid were used in these two experiments to cover the surface of the catalyst with acid in order

Exp.No.	Solvent		Catalyst				Weight of linoleic acid 65 % [g]	Hydrogen volume [mL]	Temp. [°C]	Pressure [10 ⁵ Pa]	Reaction time [min]
	Volume [mL]	Type	Weight [g]	Pd content %	Substrate	Catalyst type					
1	50	I	5	0.5	α -Al ₂ O ₃	S	1	80	22	1	43
2	50	I	5	0.5	α -Al ₂ O ₃	S	2	80	22	1	33
3	50	I	5	0.5	α -Al ₂ O ₃	S	4	80	22	1	30
4	-	-	5	0.5	α -Al ₂ O ₃	S	10	40	22	1	120
5	-	-	5	0.5	α -Al ₂ O ₃	S	50	40	22	1	112
6	50	I	5	0.5	α -Al ₂ O ₃	S	1	100	22	1	63
7	50	I	5	0.5	α -Al ₂ O ₃	S	1	40	22	1	31
8	-	-	5	0.5	α -Al ₂ O ₃	S	50	160	22	1	154
9	-	-	5	0.5	α -Al ₂ O ₃	S	50	120	22	1	147
10	-	-	5	0.5	α -Al ₂ O ₃	S	50	80	22	1	137
11	50	III	5	2	α -Al ₂ O ₃	Z	1	50	22	1	76
12	50	I	5	2	α -Al ₂ O ₃	Z	1	50	22	1	44
13	50	II	5	2	α -Al ₂ O ₃	Z	1	50	22	1	34
14	75	I	20	0.5	α -Al ₂ O ₃	S	1	104	22	1	48
15	75	I	10	0.5	α -Al ₂ O ₃	S	1	104	22	1	66
16	75	I	5	0.5	α -Al ₂ O ₃	S	1	104	22	1	108

Table I

Exp. No.	Solvent		Catalyst			Weight of linoleic acid 65 % [g]	Hydrogen volume [ml]	Temp. [°C]	Pressure [10 ⁵ Pa]	Reaction time [min]
	Volume [ml]	Type	Weight [g]	Pd content %	Substrate					
17	75	I	2	5	α -Al ₂ O ₃	1	104	22	1	186
18	75	I	20	0.5	α -Al ₂ O ₃	1	104	22	1	43
19	75	I	2	0.5	α -Al ₂ O ₃	1	104	22	1	275
20 ⁺	75	I	-	0.1	Pd-powder	1	104	22	1	-
21	50	I	5	0.5	α -Al ₂ O ₃	4	80	35	1	14
22	50	I	5	0.5	α -Al ₂ O ₃	4	80	45	1	11
23	-	-	5	0.5	α -Al ₂ O ₃	50	80	35	1	13
24	-	-	5	0.5	α -Al ₂ O ₃	50	80	45	1	51
25	50	I	5	0.5	α -Al ₂ O ₃	4	80	22	5	23
26	50	I	5	0.5	α -Al ₂ O ₃	4	80	22	9	13
27	-	-	5	0.5	α -Al ₂ O ₃	50	80	22	5	66
28	-	-	5	0.5	α -Al ₂ O ₃	50	80	22	9	51
29 ⁺⁺	50	I	5	0.5	α -Al ₂ O ₃	1	1200 μ Ci T ₂	22	1	35
Semi tech. test	50	I	500	0.5	α -Al ₂ O ₃	500	400	45	1	270

Solvent I = diethylene glycol dimethyl ether; solvent II = diethylene glycol diethyl ether; Solvent III = diethylene glycol monoethyl ether.

Catalyst diameter: S = 4.0 - 5.8 mm; Q and U = 3.0 - 3.9 mm; Z = 4.3 - 5.4 mm.

⁺After 48 hours there is still 29 % of the initial quantity of hydrogen left.

⁺⁺ Tritium test

Table I Test conditions and reaction time for catalytic hydrogenation or tritiation of linoleic acid

to prevent any direct absorption of hydrogen on the catalyst surface and hence distortion of the test results.

The hydrogenation of 40 ml H₂ in Exp. 4 and 5 took 120 and 112 min, respectively. This represents a reduction of the reaction time of just 6 %. The increase of the linoleic acid concentration from 10 g to 50 g has only a very slight influence on the hydrogenation rate.

Comparison of the hydrogenation test results with and without solvent shows that the hydrogenation reaction is much faster with solvent.

3.2 Influence of the hydrogen concentration

In Exp. 6, 1 and 7 we used 100, 80 and 40 ml of hydrogen, respectively. According to Fig. 2 the hydrogenation rate increases as the amount of hydrogen, but not in proportion. The reaction times are 63, 43 and 31 min, i.e. 38 % and 103 % longer. These results indicate that hydrogenation of linoleic acid will take a long time with a low hydrogen concentration.

The influence of the hydrogen concentration on the hydrogenation activity without solvent was investigated in Exp. 5, 10, 9 and 8 for concentrations increasing from 40 ml in Exp. 5 to 80, 120 and 160 ml. The reaction times are 112, 137, 147 and 154 min, i.e. here, too, the hydrogenation time of the linoleic acid increases as the hydrogen concentration. The reaction time increases by 22 %, 31 % and 37 %, respectively. According to Exp. 8 approx. 413 Ci tritium can be irreversibly bonded to the double bond of linoleic acid in 154 min, when calculating the hydrogen as tritium.

3.3 Influence of the solvents

Experiments 11, 12 and 13 investigate the influence of different solvents. The results are represented in Fig. 3. The amount of solvent in each case was 50 ml. In Exp. 11 the polar solvent III, /4/ diethylene glycol monoethyl ether $\text{CH}_3\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{OH}$, was used, in Exp. 12 the non-polar solvent I, diethylene glycol dimethyl ether $\text{CH}_3\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{OCH}_3$; and in Exp. 13 the non-polar solvent II, diethylene glycol diethyl ether $\text{CH}_3\text{CH}_2\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_3$. From Fig. 3 it is clearly seen that the reaction is faster with non-

polar solvent (I and II). The reaction times in Exp. 11, 12 and 13 are 76, 44 and 43 min, respectively. The hydrogenation reaction with diethylene glycol diethyl ether is 2.2 times as fast as with diethylene glycol monoethyl ether, i.e. it is always advisable to use non-polar solvent. Comparison of the results of Exp. 12 and 13 (Fig. 3) shows that the reaction is the faster the larger the alkyl group. The hydrogenation time in Exp. 13 is 22 % shorter than that in Exp. 12.

3.4 Influence of the catalyst

3.4.1 Quantity of catalyst

The quantity of catalyst "S" (0.5 % Pd/ α -Al₂O₃) was varied from 20 g in Exp. 14 to 10 and 5 g in Exp. 15 and 16, respectively. Figure 4 shows the expected decrease of the reaction time as the quantity of catalyst is increased, but it is not linear. The hydrogenation times are 108, 66 and 43 min, i.e. when the quantity of catalyst is doubled in Exp. 15 and quadrupled in Exp. 14, the reaction time just decreases by 38 % and 55 %, respectively. According to Exp. 14 one can bond 268 Ci tritium to the linoleic acid in 48 min, but using a large quantity of catalyst, viz. 20 g. In practice, however, a compromise should be sought between the quantity of catalyst used, the cost and the reaction time.

3.4.2 Substrate

The influence of the substrate on the hydrogenation mechanism was investigated in a series of tests (17, 18, 19, 20), which are represented in Fig. 5.

Experiments 17 and 18 were conducted under the same experimental conditions, except that in Exp. 17 2 g of catalyst "Q" (5 % Pd/ α -Al₂O₃) was used, and in Exp. 18 20 g of "u" (0.5 % Pd/ α -Al₂O₃). The two catalysts have the same pellet shape (3.0 to 3.9 mm in diam.). The same amount of Pd was used in the two experiments, viz. 100 mg, whereas the amount of substrate in Exp. 18 was approx. ten times as large as in Exp. 17. The reaction of 104 ml H₂ with the linoleic acid in Exp. 18 took 43 min and in Exp. 17 186 min, i.e. the hydro-

genation time in Exp. 17 is 4.3 times as long as in Exp. 18. The short hydrogenation time in Exp. 18 may be due to the different amounts of substrate. Then again, the amount of substrate may not be the decisive factor, but rather the distribution of the Pd on the substrate surface, because the Pd concentration with catalyst "Q" was 5 % while with "u" it was just 0.5 %. This means that the Pd with the latter catalyst was thinly coated on the aluminium oxide surface and is therefore more effective than with catalyst "Q". Experiments 19 and 20 were conducted to clarify the situation. In Exp. 19 almost the same quantity of substrate was used as in Exp. 17. The difference between the two experiments resides in the amount of Pd and its distribution on the substrate surface. The amount of Pd in Exp. 17 is ten times as large as in Exp. 19. The hydrogenation times in Exp. 17 and 19 are 186 and 275 min, respectively, i.e. the hydrogenation time in Exp. 19 was increased by just 47 % although only one-tenth the amount of Pd was used. This result is clear proof that the distribution of the catalyst on the substrate surface is the decisive factor.

This finding was verified in Exp. 20. The catalyst here was 100 mg of pure powdered Pd without substrate. The amount of Pd is equivalent to that in Exp. 17. As can be seen from Fig. 5, the reaction is very slow. After 48 hours there is still 29 % of the initial quantity of hydrogen present. The reason for this is that owing to the heaviness of the Pd grains mere shaking of the reaction vessel is not sufficient to mix them thoroughly.

Catalysts of large surface area and low Pd concentration are thus optimum for practical application.

3.5 Temperature dependence

In several tests the temperature dependence of the hydrogenation reaction was investigated in the presence of solvent I (Exp. 3, 21 and 22) and without solvent (Exp. 10, 23 and 24). The temperature was varied from 22° C to 45° C.

From Fig. 6 it is seen that the reaction with solvent I is faster. As is known, the reaction rate increases as the temperature. A rise

in temperature from 22° C in Exp. 3 to 35° C in Exp. 21 halves the reaction time from 30 min to 14 min.

A further increase of the temperature to 45° C no longer causes any drastic reduction of the reaction time (see Exp. 22; 11 min at 45° C). Apparently, 35° C is enough to overcome the activation energy of the hydrogenation reaction quickly, while further temperature increase slows the reaction down again as a result of diffusion processes.

Increased temperature, however, is at least a problem for technical application because this also increases the vapour pressure of the solvent and linoleic acid. The extent to which higher temperatures affect the stability of tritiated stearic acid is to be investigated later.

3.6 Influence of the pressure

The last parameter to be investigated in the reaction between hydrogen and linoleic acid was the pressure. Figure 7 shows the influence of the pressure on the hydrogenation time, the pressure being varied from 1×10^5 Pa to 5×10^5 and 9×10^5 Pa. Experiments 3, 25 and 26 were conducted with solvent. Exp. 10, 27 and 28 without. According to Fig. 7 the reaction time varies from 30 min to 23 and 13 min with solvent, and from 137 to 66 and 51 min without. As the results generally show so far, the hydrogenation reaction here is also faster with solvent. In practice, however, the use of higher pressure calls for more elaborate apparatus, thus raising the cost, so that it is better to reduce the time by modifying other parameters, e.g. increasing the temperature.

3.7 Tritium test

The results of the tritium tests are shown in Fig. 8 for 1200 $\mu\text{Ci T}_2$ ($\approx 0.4651 \mu\text{l T}_2$). The activity of the tritiated acid produced quickly increases, reaching a constant value of 17 $\mu\text{Ci/ml}$ after 35 min, i.e. within the measuring accuracy the conversion process can be regarded as terminated.

3.8 Semi-technical test

The experimental conditions under which the results discussed were obtained do not, of course, correspond to those present in practice.

In particular, the gas volumes from which the tritium has to be extracted are much larger (4 to 5 orders of magnitude), whereas the amount of catalyst and the volume of linoleic acid can only be increased by about 3 orders of magnitude. Another major difference is the way the gas and liquid phases are mixed: in the laboratory tests the reaction partners are kept in close contact by continuous shaking; in a technical facility the gas is puffed into the column from below through the catalyst filling.

The concentration conditions are also different: in the laboratory tests the H_2 concentration is at least a few per cent (up to over 10 %) of the He atmosphere of the gas mice, whereas even in the event of accidental release the amount of T_2 present in the He atmosphere of a glove-box is at most a fraction of one per cent /5/ (e.g. release of 10^5 Ci T_2 in a 6 m^3 glove-box \triangleq 0.66 %). For this reason a semi-technical rig was established; Fig. 9 shows its components.

An amount of 500 g of catalyst "s" (7) was put (filling level 10 cm) into a 70 cm long reaction column (10) with an outer diameter of 9 cm and a volume of 4000 ml. The entire apparatus was then evacuated and filled with 1×10^5 Pa He. A mixture of 500 ml diethylene glycol dimethyl ether and 500 ml linoleic acid (65 %) was filled into the column via the filler vent (4) by means of the hose pump (5) (throughput 12 l/h) and further circulated. The column was heated to 45°C with strip heaters. A 35 l test volume (9) simulates the glove-box volume; by means of the diaphragm compressor (1) the He in this volume was circulated counter to the liquid flow via the catalyst filling. The gas flow was set at 40 l/min and monitored with the throughput gauge (8). The liquid droplets borne by the gas flow were largely retained in the separator (3), the rest being caught in the cooling trap (2). A volume of 400 ml H_2 (\triangleq 1032 Ci T_2) was then injected through the septum (6) into the gas flow. Gas samples were then taken through the same septum at defined intervals and the

H concentration was determined by gas chromatography.

The results are presented in Fig. 8. It is shown that hydrogenation occurs very fast within a time of 105 min; over 90 % of the hydrogen is used up in the process. The remaining hydrogen is then bonded to the linoleic acid after 153 min. It thus takes a total of over 4 hours (258 min) for all of the hydrogen to be bonded by the linoleic acid.

The amount of hydrogen injected relative to the total volume was just 1 %; of this >90 % (i.e. $[H_2] = 0.9 \%$) was quickly bonded. This result agrees with that of the gas mouse tests and, when extended to a technical detritiation plant, is to be regarded as the processing of an accidental tritium release ($[T_2] \sim 1 \%$). The slower reaction in the region of low H_2 concentrations would then be equivalent to a routine release (leakage, permeation of T_2), for the processing of which longer times in the region of several hours seem acceptable. It can thus be stated that a technical detritiation plant can be designed on the basis of the semi-technical test rig.

4. Summary

This report deals with investigations of heterogeneous hydrogenation (tritiation) of linoleic acid on Pd catalysts. The process is to be used for chemical bonding and hence efficient removal of the tritium accidentally released in glove-boxes.

The reaction times, i.e. the rate at which the H_2 or T_2 reacts with the linoleic acid, depends on several parameters; the investigations were concerned with the H_2 (T_2) concentrations

- the quantity of linoleic acid released (1 to 50 g)
- the temperature (22 to 45° C)
- the pressure (1 to 9×10^5 Pa)
- the solvents
- the catalyst.

Depending on the experimental conditions, reaction times of between 34 and 476 min are obtained when 0.5 to 160 ml H_2 is converted with linoleic acid. In order to scale the laboratory results better to

practical conditions, a semi-technical test was also conducted. This involved converting up to 400 ml H₂ with 500 g of linoleic acid and 500 g of Pd catalyst. The bulk of the hydrogen used (>90 %) was converted within 105 min; if the H₂ volume is taken to be tritium (>1000 Ci), more than 90 % of any tritium accidentally released could be removed from the glove-box atmosphere in under two hours.

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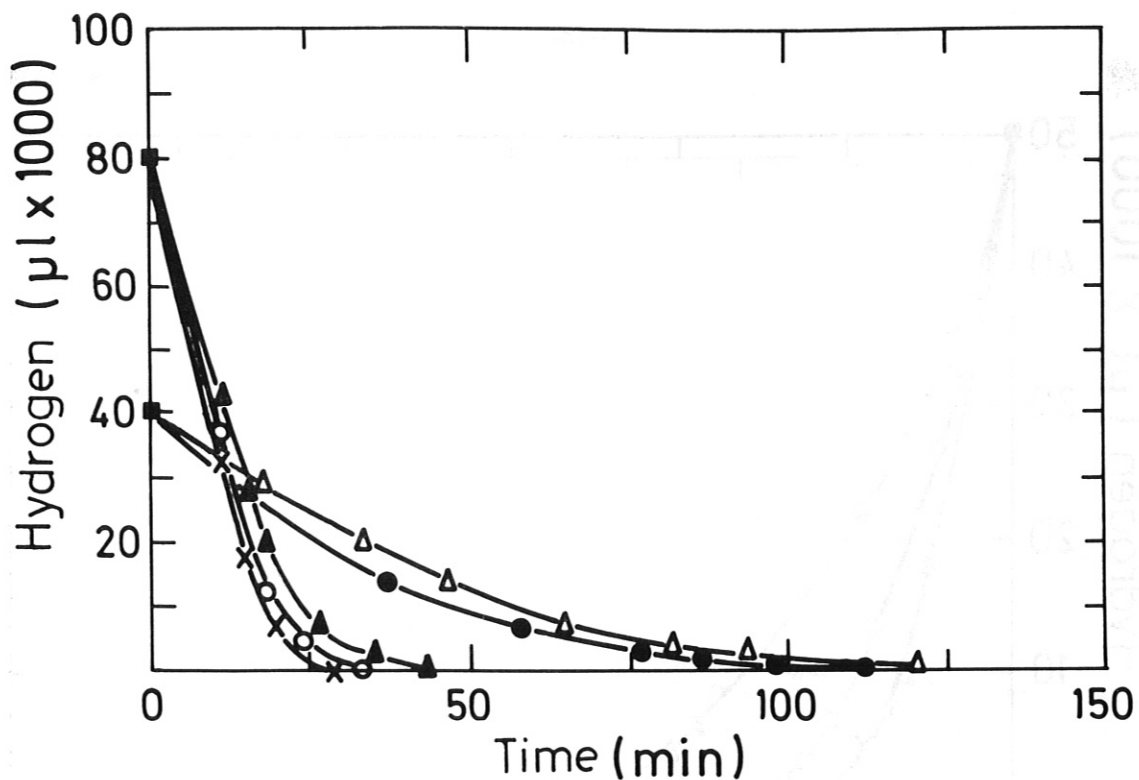


Fig. 1 Influence of the linoleic acid concentration on the reaction rate
 - 50 ml solvent I, 5 g catalyst "s", temperature 22°C , pressure 10^5 Pa
 80 ml H_2 , linoleic acid 65% (▲ 1 g, ○ 2 g, X 4 g)
 - 5 g catalyst "s", 40 ml H_2 , temperature 22°C , pressure 10^5 Pa,
 linoleic acid 65% (Δ 10 g, ● 50 g)

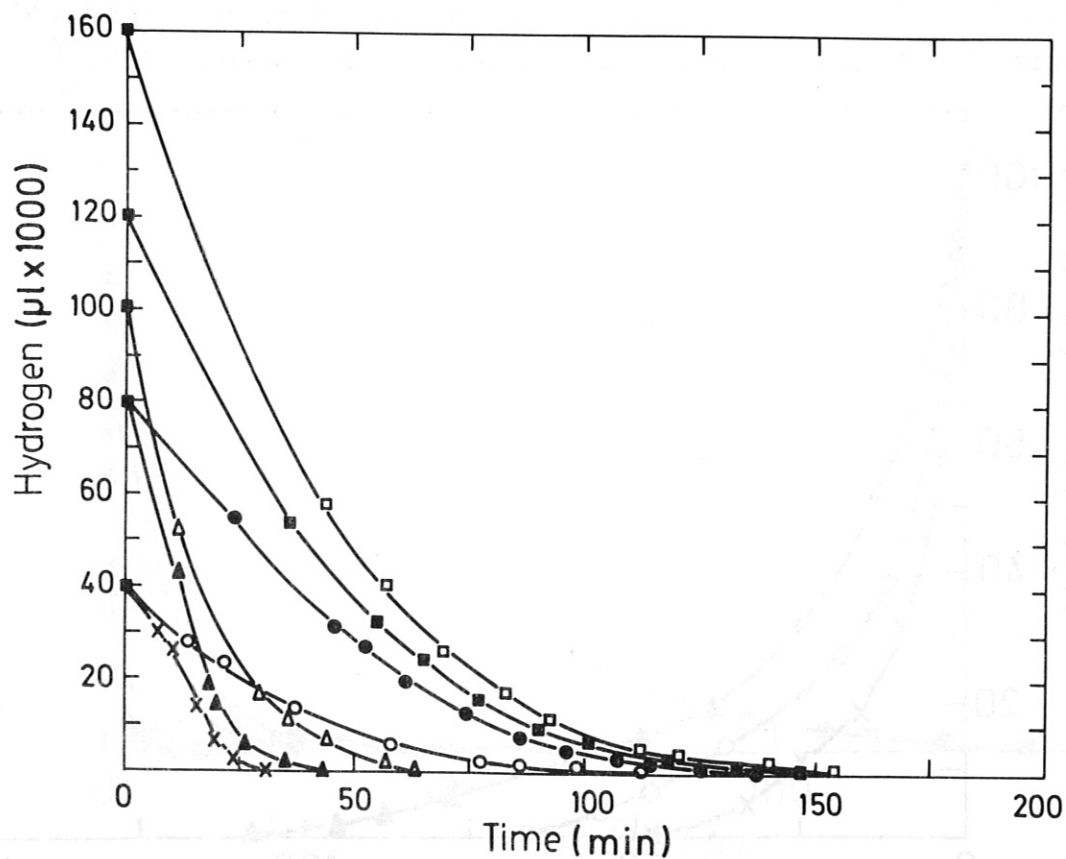


Fig. 2 Influence of the hydrogen concentration on the reaction rate
 - 50 ml solvent I, 5 g catalyst "s", 1 g linoleic acid 65%, temperature
 22°C , pressure 10^5 Pa, hydrogen (Δ 100 ml, ▲ 80 ml, X 40 ml)
 - 5 g catalyst "s", 50 g linoleic acid 65%, temperature 22°C ,
 pressure 10^5 Pa, hydrogen (□ 160 ml, ■ 120 ml, ● 80 ml, ○ 40 ml)

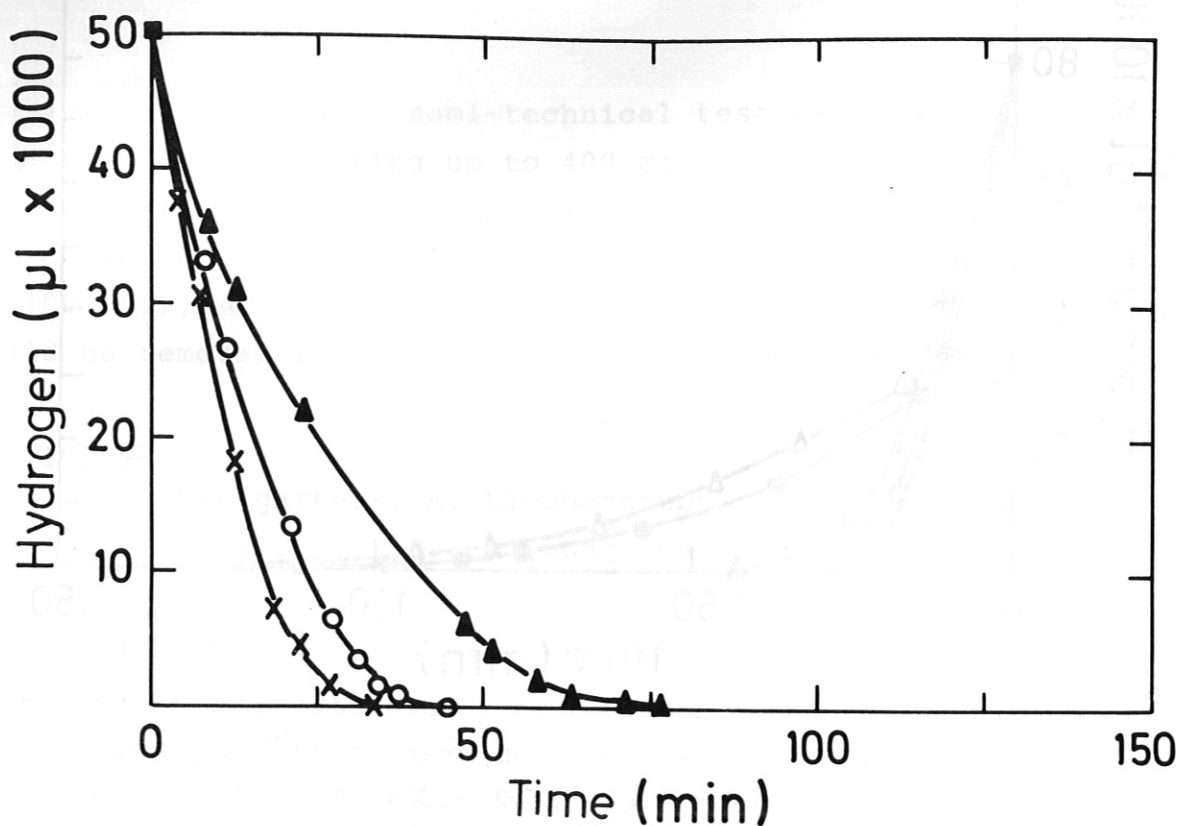


Fig. 3 Influence of the solvent on the reaction rate
- 5 g catalyst "z", 1 g linoleic acid 65 %, 50 ml H₂, temperature 22° C, pressure 10⁵ Pa, (O solvent I, X solvent II, ▲ solvent III)

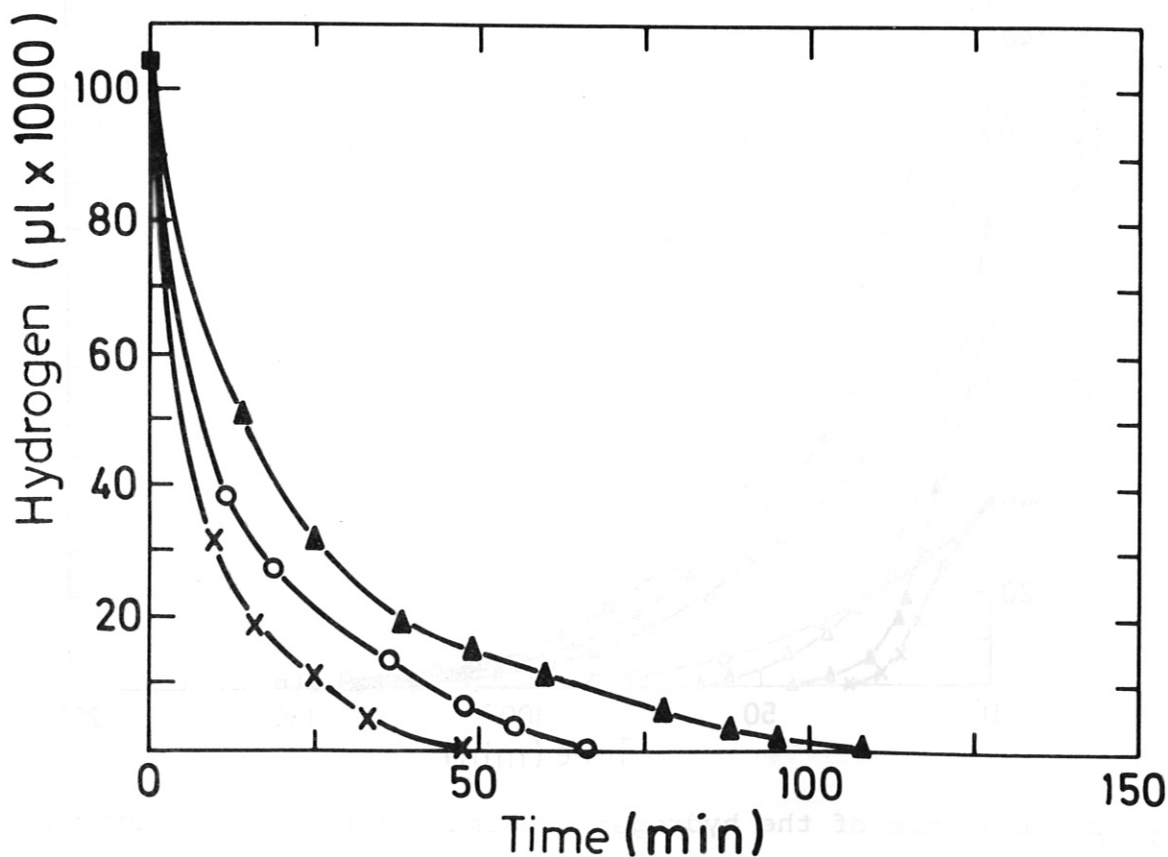


Fig. 4 Influence of the quantity of catalyst on the reaction rate
- 75 ml solvent I, 1 g linoleic acid 65 %, 104 ml H₂, temperature 22° C, pressure 10⁵ Pa, catalyst "s" (X 20 g, O 10 g, ▲ 5 g)

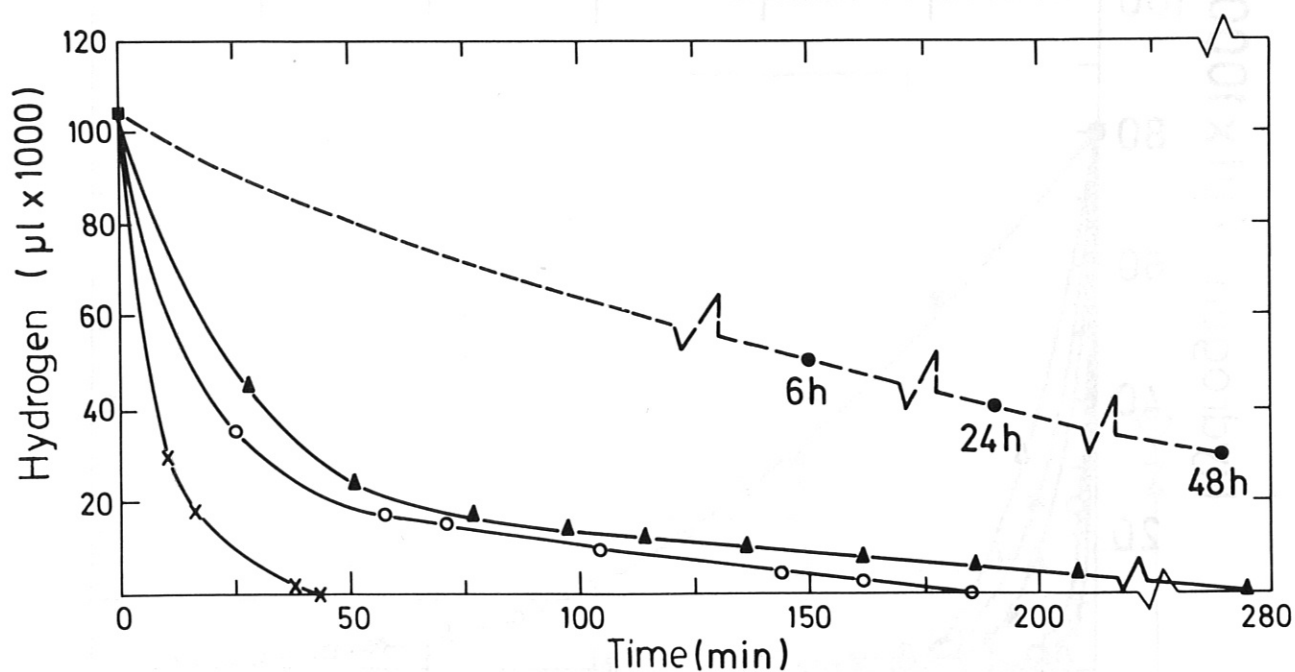


Fig. 5 Influence of the substrate on the reaction rate
 - 75 ml solvent I, 1 g linoleic acid 65 %, 104 ml H₂, temperature 22° C,
 pressure 10⁵ Pa, catalyst (○ 2 g catalyst "Q", X 20 g catalyst "u",
 ▲ 2 g catalyst "u", ● 100 mg Pd-powder)

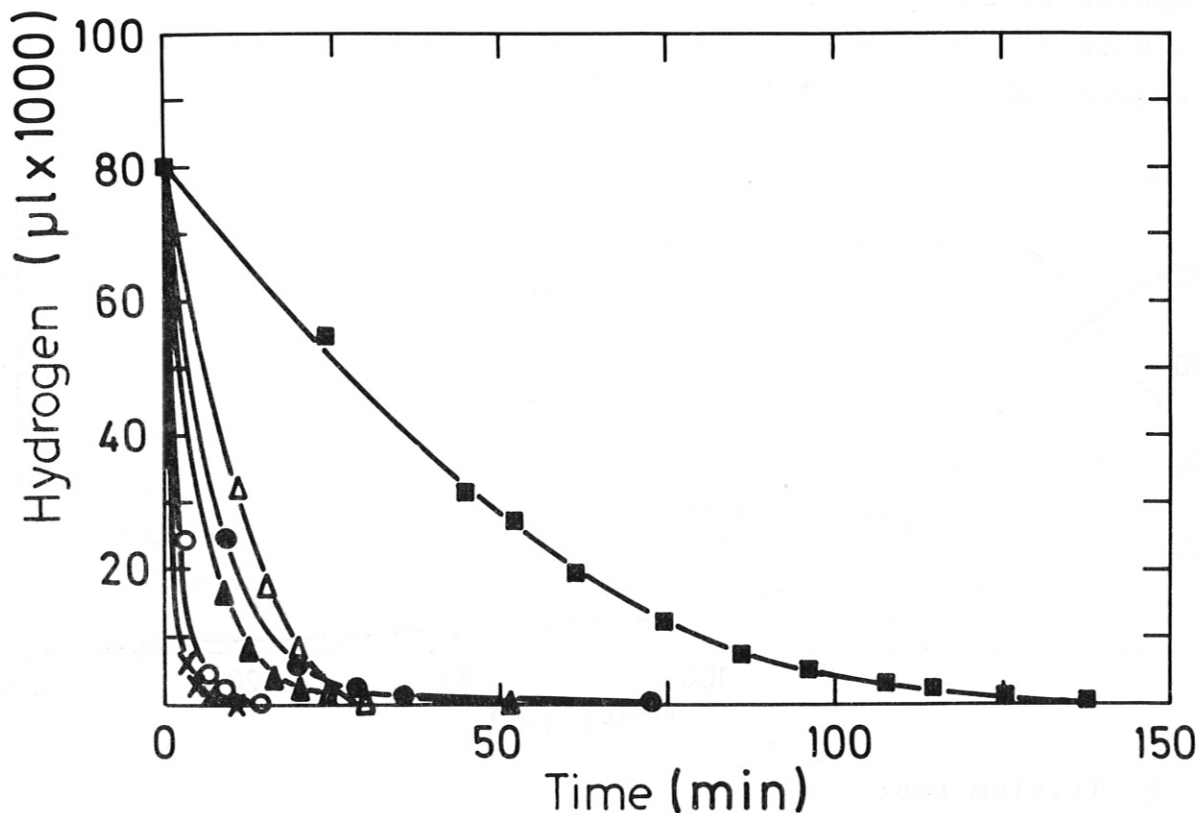


Fig. 6 Influence of the temperature on the reaction rate
 - 50 ml solvent I, 5 g catalyst "s", 4 g linoleic acid 65 %, 80 ml H₂,
 pressure 10⁵ Pa, temperature (Δ 22° C, ○ 35° C, X 45° C)
 - 5 g catalyst "s", 50 g linoleic acid 65 %, 80 ml H₂, pressure 10⁵ Pa,
 temperature (■ 22° C, ● 35° C, ▲ 45° C)

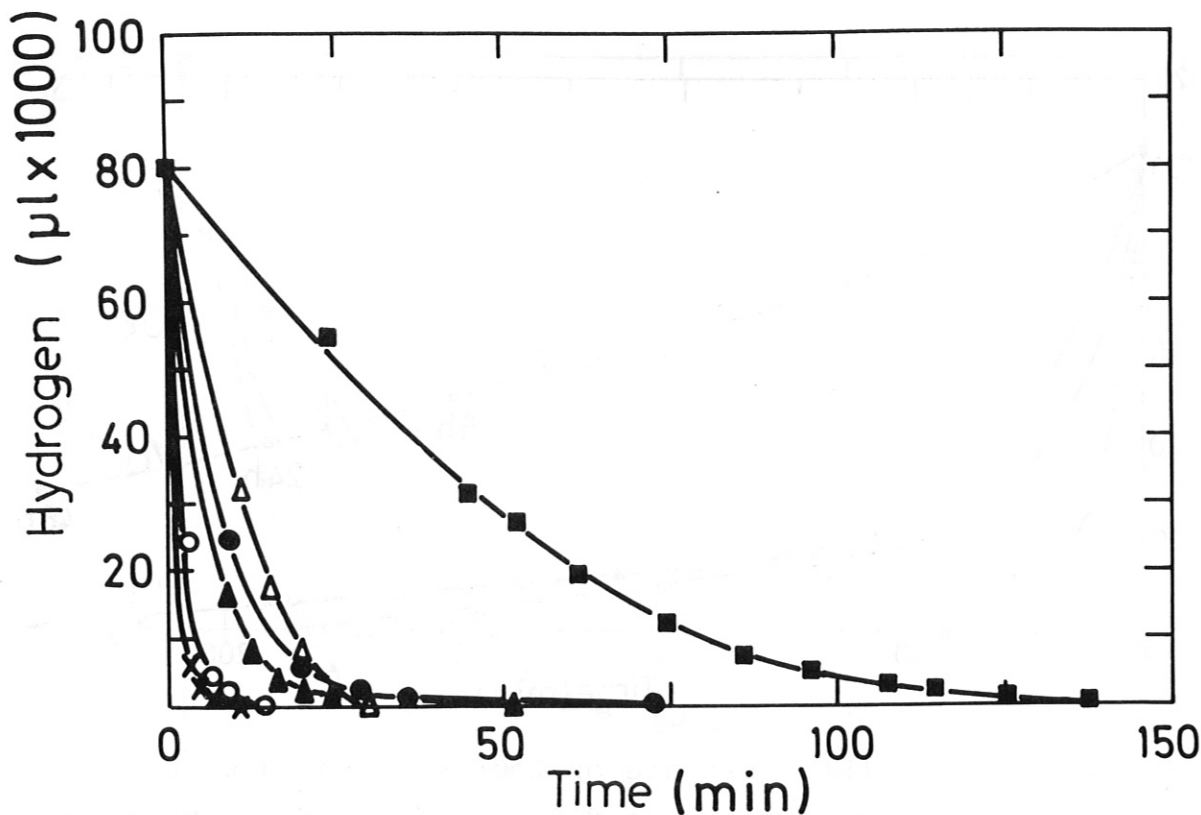


Fig. 7 Influence of the pressure on the reaction rate

- 50 ml solvent I, 5 g catalyst "s", 4 g linoleic acid 65 %, 80 ml H₂, temperature 22° C, pressure (Δ 1x10⁵ Pa, O 5x10⁵ Pa, X 9x10⁵ Pa)
- 5 g catalyst "s", 50 g linoleic acid 65 %, 80 ml H₂, temperature 22° C, pressure (■ 1x10⁵ Pa, ● 5x10⁵ Pa, ▲ 9x10⁵ Pa)

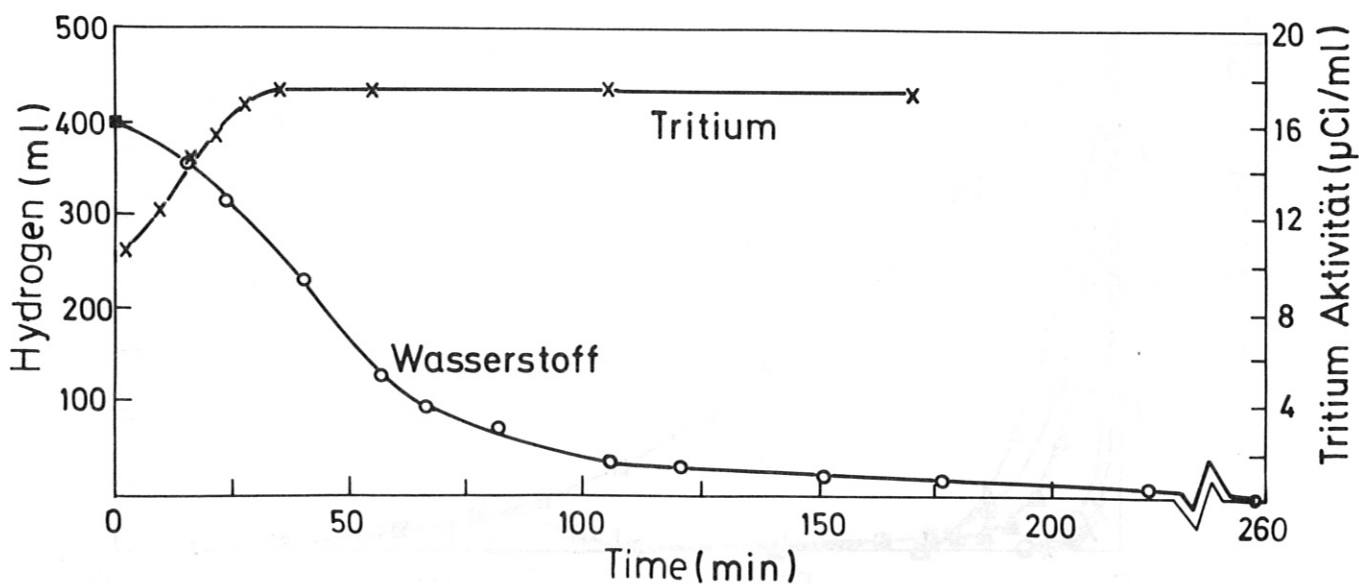
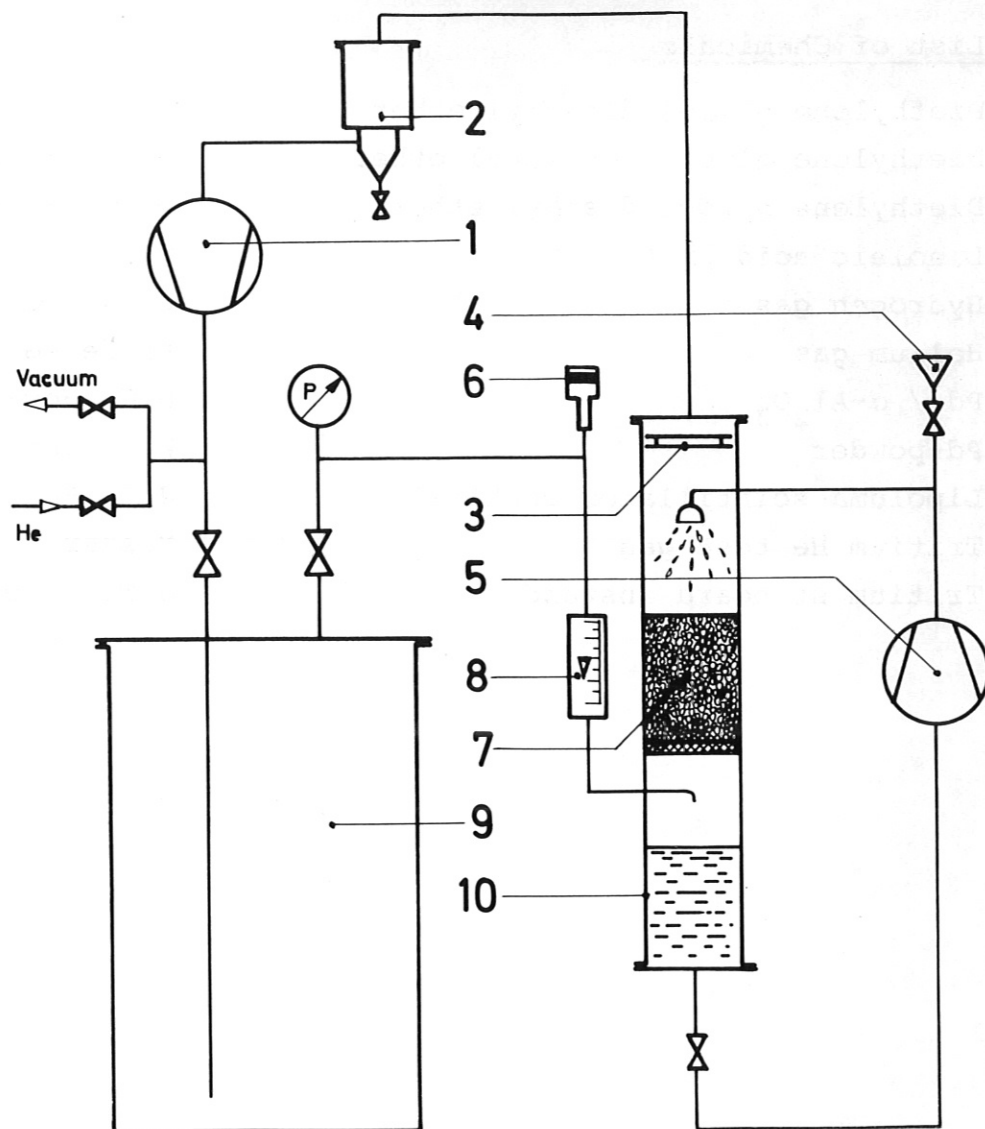


Fig. 8 Tritium test

50 ml solvent I, 5 g catalyst "s", 1 g linoleic acid 65 %, 1200 µCi T₂, temperature 22° C, pressure 10⁵ Pa

Semi-technical test

500 ml solvent I, 500 g catalyst "s", 500 g linoleic acid 65 %, 400 ml H₂, Temperature 45° C, pressure 10⁵ Pa



- | | | | |
|---|----------------------|----|------------------|
| 1 | Diaphragm compressor | 6 | Septum |
| 2 | Cooling trap | 7 | Catalyst |
| 3 | Droplet separator | 8 | Throughput gauge |
| 4 | Filler vent | 9 | Test volume |
| 5 | Hose pump | 10 | Reaction column |

Fig. 9

List of Chemicals

Diethylene glycol dimethyl ether
Diethylene glycol monoethyl ether
Diethylene glycol diethyl ether
Linoleic acid 65 %
Hydrogen gas
Helium gas
Pd / α -Al₂O₃
Pd-powder
Lipoluma scintillator cocktail
Tritium He test gas
Tritium standard instand

Suppliers

Merck-Schuchardt
Merck-Schuchardt
Merck-Schuchardt
Carl Roth KG
Linde AG
Linde AG
W.C. Heraeus GmbH
W.C. Heraeus GmbH
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