

INVESTIGATION OF THE EFFICIENCY OF INDUSTRIAL DEGREASING METHODS BY RADIOISOTOPE TRACER TECHNIQUE, II

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In a previous paper [1] we reported that the labelling of unsaturated oils with active iodine (^{131}I) by a simple method, which can be carried out with relative ease, results in an indicator suitable for the evaluation of the efficiency of various methods used for the degreasing of metal surfaces.

Tracing of grease impurities with ^{65}Zn -naphthenate

It has been further attempted to use metal soaps for the indication of grease impurities. In these investigations the isotope ^{65}Zn was found to be very advantageous from the point of view of measuring techniques. The isotope was used in the form of zinc stearate and zinc naphthenate, mixed to the grease to be labelled.

Preparation of the zinc naphthenate

^{65}Zn -naphthenate used as tracer was prepared from naphthenic acid. Solution A: 1.5 g of zinc oxide of an activity of 5 mC was suspended in the presence of 1.5 ml triethanolamine emulgator in 50 ml of water. Solution B: 2.5 g of naphthenic acid was suspended in the presence of 2 ml of triethanolamine emulgator in 150 ml of water.

Next, solutions A and B were admixed in several portions during 10 minutes at 70 °C.

Zn-naphthenate, forming a rubbery precipitate, was filtered, dried, dissolved in benzene, and again filtered. On expelling the benzene from the filtrate, an active zinc naphthenate, suitable for tracing, was obtained.

Melted and homogenized with the grease or oil to be traced, zinc naphthenate was applied in a ratio of about: 1 : 10 as indicator. On investigating the homogeneity of vaseline labelled with zinc naphthenate, the specific intensity was determined on different parts of the grease sample. Measured data (Table I) show that the distribution of the tracer substance in the vaseline is of adequate uniformity.

Table I

| Plate No. | Weight of labelled grease (g/12.2 cm ²) | Average intensity (cpm) | Specific intensity (cpm/g) |
|-----------|---|-------------------------|----------------------------|
| 1 | 0.1602 | 14,033 | 87,600 |
| 2 | 0.2380 | 20,935 | 87,960 |
| 3 | 0.3212 | 28,140 | 87,600 |

To ascertain its suitability as indicator for following the degreasing process, we investigated whether the specific intensity of the grease containing the zinc naphthenate tracer does not change during the degreasing process.

Steel sheets of given geometrical surface were coated with weighed quantities of radioindicated vaseline and with cooling-cutting oil, and the activity of the "grease impurity" was measured. Next, the sheets were placed into various solvents (benzene, benzine, carbon tetrachloride) and alkaline degreasing baths, to partially remove the superficial grease layer.

Washing time was selected so that a measurable quantity of labelled grease should remain on the surface. After partial degreasing, the specific intensity (that is to say the ratio of count and weight) was determined. This operation was repeated several times. Data measured in experiments with various solvents are shown in Tables II—VI.

Table II

| Type of grease: vaseline Isotope tracer compound: ⁶⁵ Zn-naphthenate | | Degreasing agent: benzene | |
|--|---|---------------------------|----------------------------|
| Degreasing time (sec) | Weight of the labelled grease (g/12.2 cm ²) | Measured intensity (cpm) | Specific intensity (cpm/g) |
| — | 0.0843 | 603,500 | 7.15 · 10 ⁶ |
| 20 | 0.0724 | 524,500 | 7.25 · 10 ⁶ |
| 40 | 0.0564 | 405,000 | 7.20 · 10 ⁶ |
| 100 | 0.0365 | 261,200 | 7.15 · 10 ⁶ |
| 160 | 0.0179 | 130,900 | 7.32 · 10 ⁵ |

Table III

| Type of grease: vaseline Isotope tracer compound: ⁶⁵ Zn-naphthenate | | Degreasing agent: Carbon tetrachloride | |
|--|---|---|----------------------------|
| Degreasing time (sec) | Weight of the labelled grease (g/12.2 cm ²) | Measured intensity (cpm) | Specific intensity (cpm/g) |
| — | 0.0667 | 544,000 | 8.15 · 10 ⁶ |
| 20 | 0.0360 | 295,400 | 8.20 · 10 ⁶ |
| 40 | 0.0141 | 113,000 | 8.02 · 10 ⁶ |

Table IV

| Type of grease: vaseline Isotope tracer compound: ⁶⁵ Zn-naphthenate | | Degreasing agent: alkaline bath | |
|--|---|------------------------------------|----------------------------|
| Degreasing time (sec) | Weight of the labelled grease (g/12.2 cm ²) | Measured intensity (cpm) | Specific intensity (cpm/g) |
| — | 0.0702 | 572,000 | 8.15 · 10 ⁶ |
| 10 | 0.0437 | 363,000 | 8.30 · 10 ⁶ |
| 15 | 0.0367 | 302,800 | 8.25 · 10 ⁶ |
| 20 | 0.0202 | 167,800 | 8.30 · 10 ⁶ |

Table V

| Type of grease: cooling-cutting oil Isotope tracer compound: ⁶⁵ Zn-naphthenate | | Degreasing agent: benzene | |
|---|---|---------------------------|----------------------------|
| Degreasing time (sec) | Weight of the labelled grease (g/12.2 cm ²) | Measured intensity (cpm) | Specific intensity (cpm/g) |
| — | 0.0297 | 236,000 | 2.55 · 10 ⁶ |
| 10 | 0.0581 | 152,500 | 2.62 · 10 ⁶ |
| 20 | 0.0266 | 70,700 | 2.66 · 10 ⁶ |

Table VI

| Type of grease: cooling-cutting oil Isotope tracer compound: ⁶⁵ Zn-naphthenate | | Degreasing agent: petrol | |
|---|---|--------------------------|----------------------------|
| Degreasing time (sec) | Weight of the labelled grease (g/12.2 cm ²) | Measured intensity (cpm) | Specific intensity (cpm/g) |
| — | 0.1000 | 304,400 | 3.00 · 10 ⁶ |
| 10 | 0.0602 | 186,000 | 3.10 · 10 ⁶ |
| 20 | 0.0089 | 27,000 | 3.05 · 10 ⁶ |

It can be seen from the results that the specific intensity of the labelled greases remained constant within ± 10 per cent during the degreasing processes.

Thus, ⁶⁵Zn-naphthenate, similarly to iodinated oils, proved to be a suitable radioactive indicator. (Cathodic degreasing in alkaline media forms an exception!)

The behaviour of thin, tracer-containing grease layers on metal surfaces during degreasing

In case of thin grease layers which cannot be followed by weighing (where the constancy of the specific intensity cannot be proved), the suitability of the indicator was investigated as follows:

The grease to be traced was labelled with iodinated linseed oil and zinc naphthenate. It is to be assumed that when the ratio of the two indicator

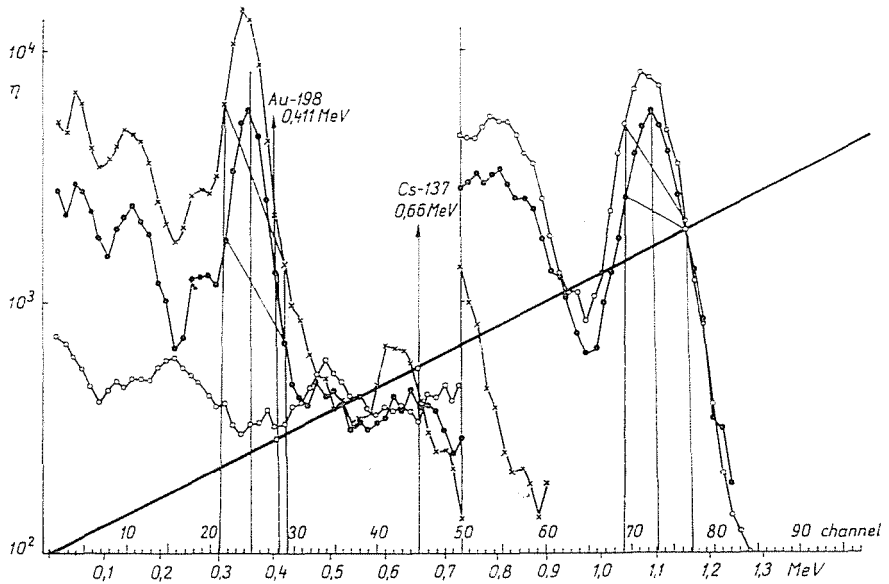


Fig. 1. ^{131}I $\times-\times$. ^{65}Zn $\circ-\circ$. $^{131}\text{I} + ^{65}\text{Zn}$ $\bullet-\bullet$

Apparatus: single channel analyser. $H\nu$: $d = 2$; $f = 8$. Gain = 10 \times . Channel width: 1 V. Time: 6 sec

compounds of different types (^{131}I -linseed oil and ^{65}Zn -naphthenate) does not change during degreasing, the quantity of the traced greases (e. g. rapeseed oil, cutting oil, vaseline) will also decrease at the same ratio, and the process is adequately traced by the indicators.

In the knowledge of the individual gamma-spectra of ^{131}I and ^{65}Zn and the spectrum of their mixtures (Fig. 1), those two spectrum intervals have been determined, at which ^{131}I and ^{65}Zn intensities can be measured unequivocally. These were found to be the photo-peaks corresponding to a gamma-energy of 0.36 MeV in case of ^{131}I and to 1.11 MeV in case of ^{65}Zn . For ^{65}Zn , the intensity of the photo-peak is to be considered in first approximation as proportional to the quantity of the isotope, without the need of any correction. However, the 0.36 MeV peak of ^{131}I is superponed onto the Compton-

region of ^{65}Zn , necessitating a correction. For this purpose, the simplified method of COVELL [2] proved suitable.

After certain degreasing periods, the isotope ^{131}I was determined in the interval corresponding to 0.36 MeV, the isotope ^{65}Zn in the discriminator voltage range corresponding to 1.1 MeV, at a channel width of 9 V. Measuring results are shown in Tables VII—XII. In view of the mechanism of degreasing processes, it may be assumed that up to a monomolecular layer, the ratio of the traced greases and the indicator compounds remains practically constant. This conclusion was also reached on comparing the behaviour — the rate and the efficiency of degreasing — of the greases labelled as described above with the dissolution of stearic acid labelled with ^{14}C isotope.

Table VII

| Type of grease: vaseline Isotope tracer compounds: ^{131}I -linseed oil and ^{65}Zn - naphthenate | | Degreasing agent: trichloro ethylene | | |
|--|--------------------|---|--------------------|-----------------------------|
| Degreasing time (min) | Trace isotope | | | |
| | ^{131}I | | ^{65}Zn | |
| | Intensity (cpm) | Residual quantity % | Intensity (cpm) | Residual quantity (%) |
| — | 129,450 | 100 | 84,880 | 100 |
| 0.5 | 57,834 | 44.5 | 34,480 | 40.6 |
| 1.5 | 400 | 0.3 | 255 | 0.3 |
| 4.5 | 80 | 0.06 | 102 | 0.12 |

Table VIII

| Type of grease: vaseline Isotope tracer compounds: ^{131}I -linseed oil and ^{65}Zn - naphthenate | | Degreasing agent: NaOH- solution, 200 g/lit. at 160 °C | | |
|--|--------------------|---|--------------------|-----------------------------|
| Degreasing time (min) | ^{131}I | | ^{65}Zn | |
| | Intensity (cpm) | Residual quantity (%) | Intensity (cpm) | Residual quantity (%) |
| | — | 432,852 | 100 | 91,770 |
| 0.5 | 153 | 0.04 | 26 | 0.03 |

Table IX

| Type of grease: vaseline Isotope tracer compounds: ^{131}I -linseed oil and ^{65}Zn - naphthenate | | Degreasing agent: NaOH- solution, 100 g/lit., 90 °C, and electrolytic degreasing | | |
|--|---------------------------|--|--------------------|-----------------------------|
| Degreasing time (min) | T r a c e r i s o t o p e | | | |
| | ^{131}I | | ^{65}Zn | |
| | Intensity (cpm) | Residual quantity (%) | Intensity (cpm) | Residual quantity (%) |
| — | 690,400 | 100 | 32,790 | 100 |
| 0.3 | 4,300 | 0.62 | 128 | 0.40 |
| 3.3 | 935 | 0.14 | 50 | 0.15 |
| 0.2 | 240 | 0.035 | 5 | 0.015 |

Table X

| Type of grease: stearic acid Isotope tracer compounds: ^{131}I -linseed oil and ^{65}Zn - naphthenate | | Degreasing agent: Trichloro ethylene and electrolytic degreasing | | |
|--|---------------------------|--|--------------------|-----------------------------|
| Degreasing time (min) | T r a c e r i s o t o p e | | | |
| | ^{131}I | | ^{65}Zn | |
| | Intensity (cpm) | Residual quantity (%) | Intensity (cpm) | Residual quantity (%) |
| — | 596,693 | 100 | 62,410 | 100 |
| 0.5 | 377,420 | 63.2 | 52,760 | 84.8 |
| 1.0 | 110,537 | 18.5 | 16,910 | 27.3 |
| 5.0 | 2,600 | 0.44 | 204 | 0.33 |
| 9.5 | 2,351 | 0.39 | 198 | 0.31 |
| 17.0 | 910 | 0.15 | 110 | 0.17 |
| 57.0 | 293 | 0.05 | 21 | 0.03 |
| 1.0 | 159 | 0.03 | 18 | 0.03 |

Efficiency and performance of degreasing solvents and alkaline degreasing baths

The isotope tracer technique described above was applied for the evaluation of the degreasing efficiency of a few organic solvents and alkaline degreasing baths.

In our investigations the following "grease impurities" were used to study their removability:

- rapeseed oil (glycerine ester) labelled with ^{131}I and ^{65}Zn -naphthenate,
- cooling-cutting oil (liquid paraffine hydrocarbons)

Table XI

| Type of grease: stearic acid Isotope tracer compounds: ¹³¹ I-linseed oil and ⁶⁵ Zn- naphthenate | | Degreasing agent: NaOH- solution, 100 g/lit., 100 °C, and electrolytic degreasing | | |
|--|--------------------|---|--------------------|-----------------------------|
| Degreasing time (min) | Tracer isotope | | | |
| | ¹³¹ I | | ⁶⁵ Zn | |
| | Intensity (cpm) | Residual quantity (%) | Intensity (cpm) | Residual quantity (%) |
| — | 800,650 | 100 | 91,809 | 100 |
| 2 | 252,400 | 31.2 | 15,500 | 16.9 |
| 3 | 8,147 | 1.00 | 727 | 0.79 |
| 11 | 3,068 | 0.38 | 658 | 0.72 |
| 2 | 1,582 | 0.20 | 375 | 0.40 |

Table XII

| Type of grease: stearic acid Isotope tracer compounds: ¹³¹ I-linseed oil and ⁶⁵ Zn- naphthenate | | Degreasing agent: 30% Na ₂ SiO ₃ -solution; 75 °C | | |
|--|--------------------|--|--------------------|-----------------------------|
| Degreasing time (min) | Tracer isotope | | | |
| | ¹³¹ I | | ⁶⁵ Zn | |
| | Intensity (cpm) | Residual quantity (%) | Intensity (cpm) | Residual quantity (%) |
| — | 1,060,388 | 100 | 127,640 | 100 |
| 1.5 | 906,560 | 91 | 118,740 | 93 |
| 3.0 | 55,262 | 5.2 | 6,465 | 5.05 |
| 9.0 | 3,028 | 0.30 | 479 | 0.37 |

- stearic acid (fatty acid),
- vaseline (ointment-type paraffine hydrocarbon). (These last three greases were labelled with ⁶⁵Zn-naphthenate.)

Degreasing with organic solvents

The efficiency of organic solvents used in practice, thus that of benzine, benzene, trichloroethylene, carbon tetrachloride and ethyl alcohol, was investigated. Tests were carried out on deep-drawn steel plates of 4 × 4 cm, artificially contaminated. The quantity of grease on the surface of the plates was determined by measuring the activity. Next, the plates were placed for a known time into the solvent. After this period, the plate was taken

out of the solvent, and the average quantity of the grease, remaining on the surface, was determined by again measuring the activity. This procedure was repeated until the quantity of the grease remaining on the surface was reduced no further.

The decrease in the quantity of grease attained by the degreasing with solvents is plotted on diagrams. Degreasing in various solvents is shown by Figs 2—4. Fig. 5 shows the results of three parallel experiments carried out

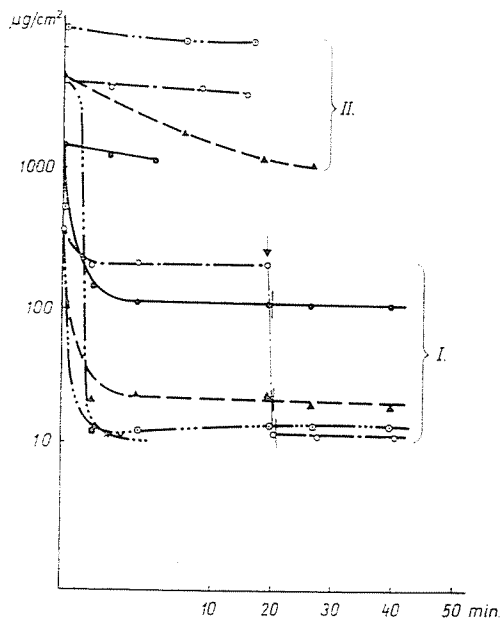


Fig. 2. Solvents: I. Benzene. II. Ethyl alcohol
 Temperature: 20 °C. Greases: Rapeseed oil ●—● Cooling-cutting oil ▲—▲
 Vaseline ○—○ Linseed oil ○—○ Stearic acid ○—○

in 50 ml of carbon tetrachloride with two test plates. Figs 6 and 7 illustrate the results obtained in a three-step degreasing experiment. In these experiments, the plate to be degreased was placed into 25 ml of the solvent. When the grease on the surface of the plate was not reduced further in this solvent, the plate was transferred into the next fresh 25 ml of the solvent. When also in this step, the quantity of grease did not decrease further, degreasing was continued in a fresh 25 ml portion of the solvent. In the same solvents, further two contaminated test plates were degreased in a similar way. The figures illustrate clearly processes taking place in tree-step counter-current industrial degreasing equipment of batchwise operation. It can be seen that, even in a fresh portion of solvent, the degreasing rate decreases considerably with the decrease of the superficial grease layer.

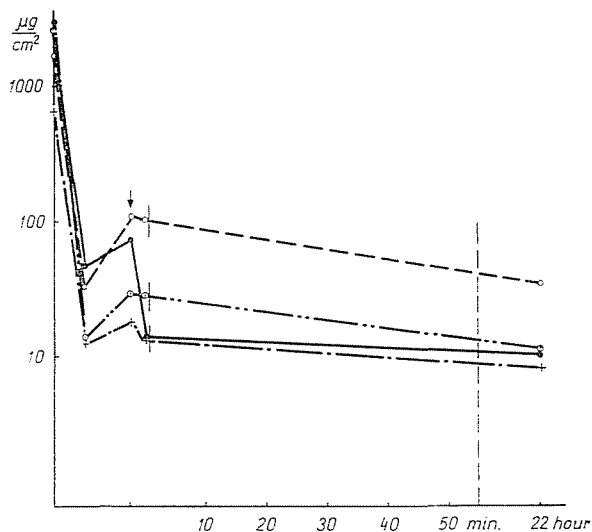


Fig. 3. Solvent: Trichloro ethylene

Temperature: 20 °C. Greases: Rapeseed oil ●—● Vaseline ○—○ Linseed oil ○---○
Stearic acid ○-·-○

Alkaline degreasing baths and the efficiency of their individual components

According to experiences gained in practice, alkaline degreasing is more economic and efficient, than degreasing with solvents. Therefore, in the further part of our experiments, these methods were studied, with particular emphasis on degreasing rate (on account of the long operational times required in practice) and on the efficiency of degreasing, to then compare experimental results with those obtained in degreasing with solvents. The usual components of the customary alkaline degreasing baths are sodium hydroxide, carbonate, phosphate, cyanide and silicates. The aqueous solutions of these alkalis — together with surface tension decreasing and emulsifying additives — constitutes the baths for the degreasing of metal surfaces. These components are compounded on empirical basis, according to a great number of formulations, by the various plants, and a great variety of bath compositions are also reported in literature. This may be ascribed to the fact that the action and the role of the single components is not completely cleared.

In our experiments also the individual efficiency of the single component has been investigated.

The efficiency of sodium hydroxide was investigated in aqueous solutions of 50, 100 and 200 g/lit. concentrations (in accordance with the concentration of degreasing baths used in practice) without additives, at 50 and 95 °C. Measuring results are shown in Figs 8—10; the quantity of grease on the

surface was plotted in units of $\mu\text{g}/\text{cm}^2$ on a logarithmic scale; on Fig. 9, results obtained in experiments with distilled water are shown as comparison.

Curves of similar shape were obtained in aqueous solutions of NaCN , Na_2SiO_3 , Na_3PO_4 and Na_2CO_3 (in our experiment no mechanical agents were

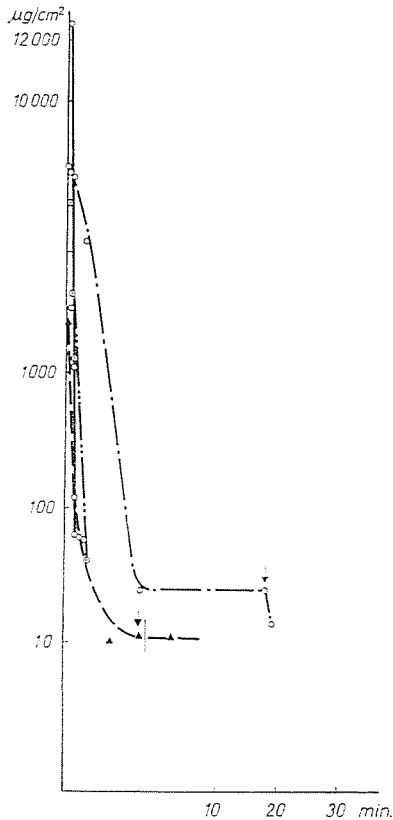


Fig. 4. Solvent: Carbon tetrachloride
 Temperature: 20 °C. Greases: Rapeseed oil ●—● Vaseline ▲--▲ Linseed oil ○- - -○ Stearic acid ○- - -○

used, the plates were immersed into resting liquid, similarly to the experiments on the degreasing with solvents).

The temperature of the solution plays a very important role in the process. Therefore, similarly to plant practice, our experiments were undertaken at 95 °C, and also further findings refer to degreasing in baths at this temperature.

On evaluating the individual efficiency of the components of degreasing baths, the following can be stated:

a) The investigated alkalies exert also alone, without wetting agents, a degreasing action.

b) From the point of view of efficiency and rate of degreasing, the aqueous solution of sodium hydroxide proved to be the best. For the investigated components, the following order can be established:

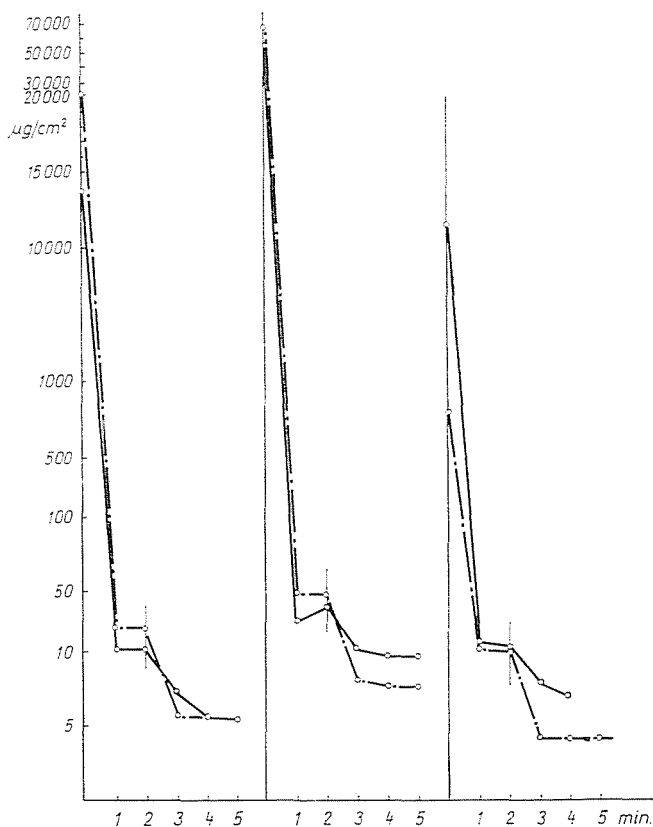


Fig. 5. Solvent: Carbon tetrachloride

Temperature: 20 °C. Volume of solvent: 50—50 ml. Grease: Linseed oil (labelled with ^{131}I)
 Degreased plates: 1. \circ — \circ (14 cm 2). 2. \circ — --- \circ (14 cm 2). (The vertical straight line indicates new, pure solvent)

- | | |
|---------------------|-------------------------------------|
| 1. NaOH | (optimum concentration: 100 g/lit.) |
| 2. NaCN | „ „ 100 g/lit.) |
| 3. Na $_2$ SiO $_3$ | „ „ 10 g/lit.) |
| 4. Na $_3$ PO $_4$ | „ „ 200 g/lit.) |
| 5. Na $_2$ CO $_3$ | „ „ 200 g/lit.) |

The above order corresponds to the decreasing order of the pH-values of the listed alkalis in a 10 per cent aqueous solution (in the range from 14 to 11.6 pH) at 20 °C.

The important role of sodium hydroxide also becomes apparent from experiments with solutions containing two or more components. It can be

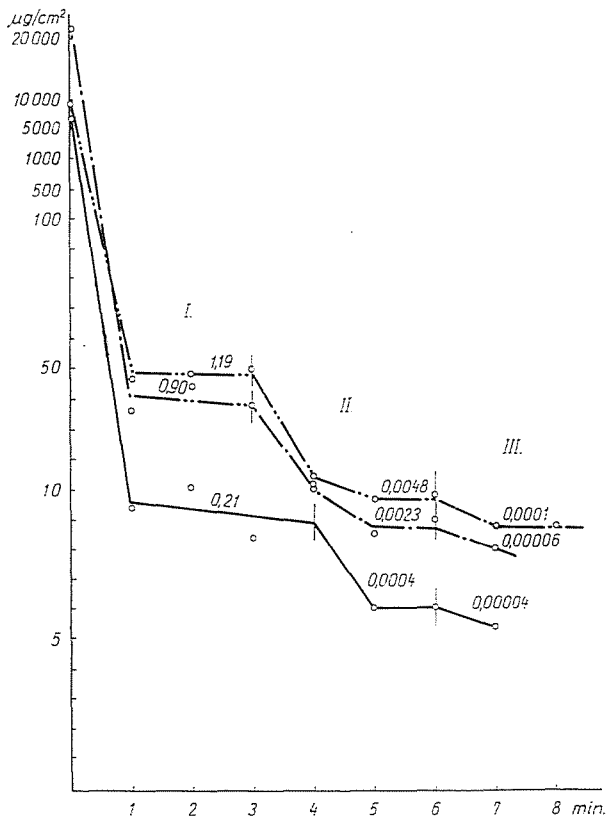


Fig. 6. Degreasing in three steps on three plates. Solvent: Carbon tetrachloride
 Temperature: 20 °C. Volume of solvent: 25–25 ml. Grease: Linseed oil. Degreased plates:
 1. ○—○ 2. ○—.—.—○ 3. ○—.—.—○ Steps: I, II, III (Numbers above the curves
 indicate the grease contamination of the solvent in g of grease per 100 ml of solvent)

seen from Fig. 11 that the solution not containing sodium hydroxide gave the lowest degreasing efficiency. (In these experiments the mixture of the four grease types mentioned above was applied as contaminant.)

The efficiency of industrial baths of various composition has also been investigated. Fig. 12 shows the degreasing process in two baths of different compositions. Degreasing proceeded at the same rate and with the same efficiency in the bath containing a wetting agent as additive and having a

surface tension of 35 dyn/cm, and in that containing no wetting agent and having a surface tension of 69 dyn/cm.

According to our observations, the rate and the efficiency of degreasing is not practically influenced by the surface tension. It was unequivocally found that efficiency is determined primarily by the pH-value of the bath.

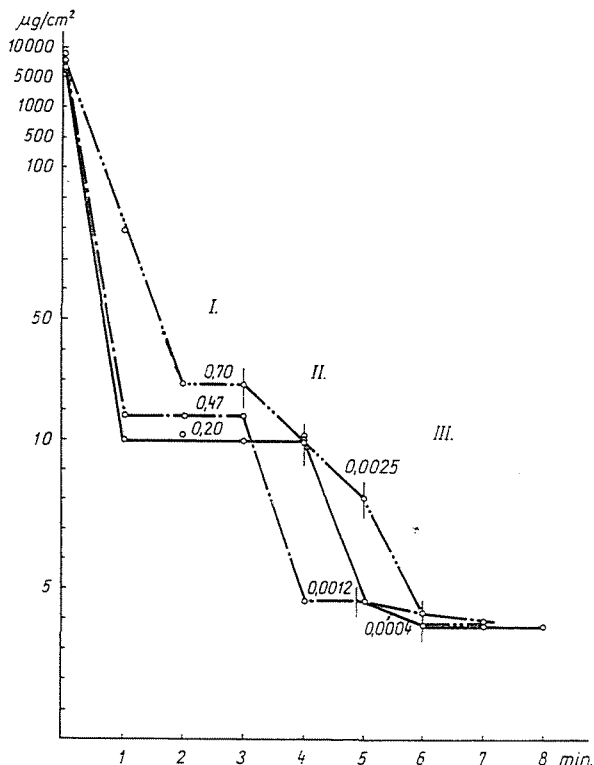


Fig. 7. Degreasing in three steps on three plates

Solvent: Benzene. Temperature: 20 °C. Volume of solvent: 25–25 ml. Grease: Linseed oil.
Symbols: identical with those used in Fig. 6

The “wetting agent”, that is to say the surface tension of the bath plays a role in the emulsification and the prevention of the reabsorption of the grease removed from the metal surface. Our experiences show the rate of degreasing to be independent of the type of the grease impurity, of its chemical composition, and, with the exception of sodium hydroxide, to a certain extent even of the nature of the components of the bath. It was found that the rate of removal of grease impurities met within practice ($10^2 - 10^3 \mu\text{g}/\text{cm}^2$) in the first 5 minutes of degreasing, during which the superficial grease impurities

are reduced to about $10 \mu\text{g}/\text{cm}^2$, can be described with an equation of the type

$$\lg G = -At + B.$$

After the first 5 minutes of the operation, the rate of degreasing decreases, and the process can be represented by the following type of equation:

$$\lg G = C/t + D,$$

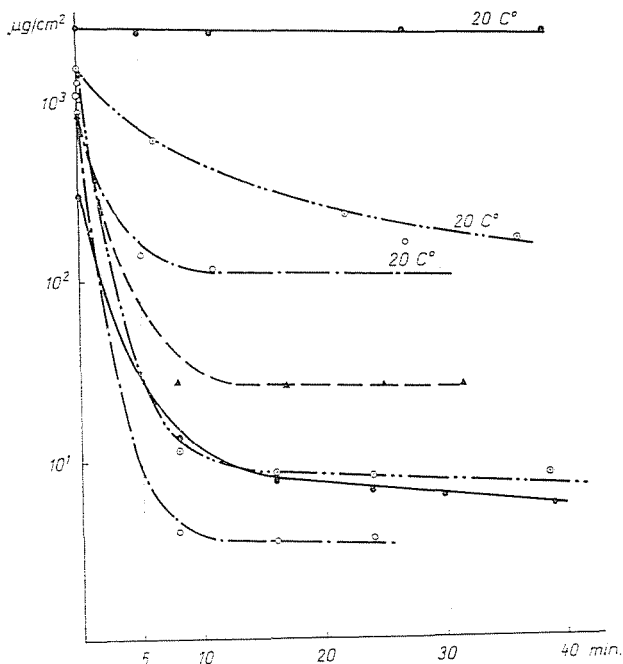


Fig. 8. Degreasing agent: Sodium hydroxide (NaOH) solution. Concentration: 50 g/lit. Temperature: 95 and 20 °C, respectively. Greases: Rapeseed oil \odot — \odot Vaseline \triangle — \triangle Linseed oil \square — \square Stearic acid \diamond — \diamond

where G is the quantity of the grease impurity on the surface ($\mu\text{g}/\text{cm}^2$), t is the time of operation (min), and A , B , C , D are constants, depending on the composition and the temperature of the degreasing bath.

(In the "standard" baths shown in Fig. 12, the rate constants are as follows: $A = 0.5$ to 1; $B = 3$ to 4; $C = 2.5$ to 4.5; $D = 0.15$ to 0.20.)

It may be stated as a requirement of alkaline degreasing baths that the efficiency should attain a value corresponding to $\lg G = 4.5/t + 0.2$, or more, that is to say, that after an operational time of 10 to 15 minutes, not more than 2 to 5 $\mu\text{g}/\text{cm}^2$ of grease impurity should remain on the surface.

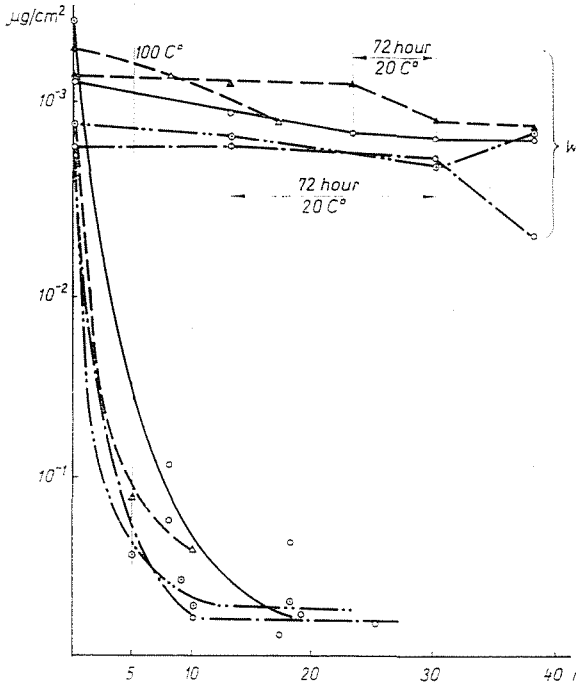


Fig. 9. Degreasing agent: Sodium hydroxide (NaOH) solution. Concentration: 100g/lit. Temperature: 95 °C. Greases: Rapeseed oil ○—○ Cooling-cutting oil ▲-----▲ Vaseline △-----△ Linseed oil ○-----○ Stearic acid ⊙-----⊙ (Curves marked with W indicate the decrease of grease quantity obtained in distilled water at 95 °C)

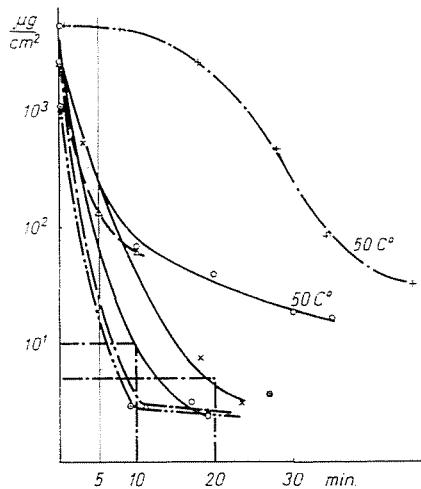


Fig. 10. Degreasing agent: Sodium hydroxide (NaOH) solution Concentration: 200 g/lit. Temperature: 95 and 50 °C, respectively Greases: Rapeseed oil ●—● Cooling-cutting oil ▲—▲ Vaseline ×—× Linseed oil ----- Stearic acid ⊙-----⊙

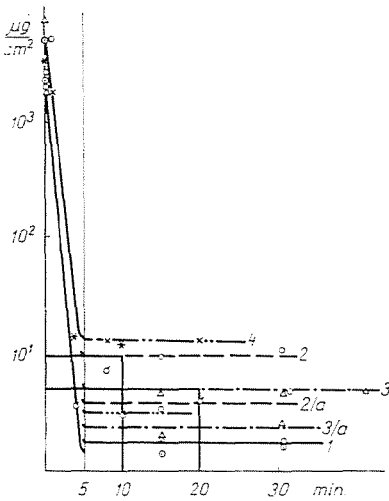


Fig. 11. Degreasing agent: Multicomponent alkali solution (1—5)

Temperature: 95 °C

Composition of the degreasing baths:

- | | |
|--|--|
| 1. NaOH 50 g/lit. | 3. NaOH 50 g/lit. |
| Na ₃ PO ₄ 50 g/lit. | Na ₂ SiO ₃ 50 g/lit. |
| 2. NaOH 25 g/lit. | 4. Na ₂ CO ₃ 50 g/lit. |
| Na ₃ PO ₄ 25 g/lit. | Na ₃ PO ₄ 50 g/lit. |
| 5(2/a, 3/a) NaOH 25 g/lit. | |
| Na ₂ CO ₃ 25 g/lit. | |
| Na ₃ PO ₄ 25 g/lit. | |
| Na ₂ SiO ₃ 25 g/lit. | |

Symbols: --- Vaseline
 ——— Rapeseed oil
 - - - Linseed oil
 ····· Stearic acid

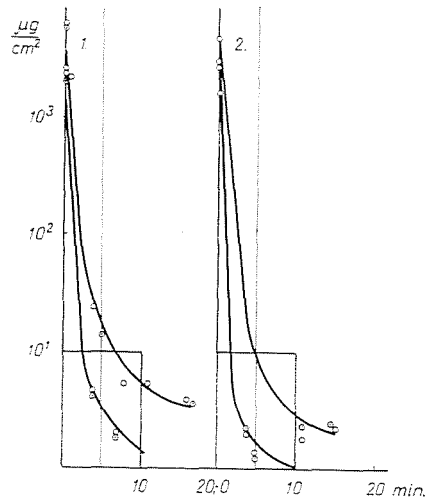


Fig. 12. Degreasing agent: Alkaline industrial degreasing bath

Temperature: 95 °C

Composition of the degreasing baths:

| Components | 1 g/lit. | 2 g/lit. |
|----------------------------------|----------|----------|
| NaOH | 25 | 100 |
| Na ₃ PO ₄ | 25 | 20 |
| Na ₂ CO ₃ | 20 | 40 |
| Na ₂ SiO ₃ | 1 | 1 |

Symbols: ○—○ without the addition of a wetting agent
 ⊙—⊙ with the addition of 1g/lit. of wetting agent P.F.8

This quantity can be reduced by electrolytic degreasing. In this procedure, the work-pieces placed into the alkaline bath are connected as anode and cathode, respectively, and electrolyzed for 2 minutes. The superficial grease impurities can be reduced with this method within a relatively short time (1 to 2 minutes) to 1 to 0.5 $\mu\text{g}/\text{cm}^2$. With this method in our experiments, grease impurities could be reduced to as low a value as 0.1 $\mu\text{g}/\text{cm}^2$.

In the initial period of alkaline degreasing, the removal of the relatively thick grease layer from the metal surface is not uniform. This fact is verified also by the autoradiograms No. 1—3, showing the superficial distribution of a grease layer of an average thickness of 500—250 $\mu\text{g}/\text{cm}^2$ after a degreasing

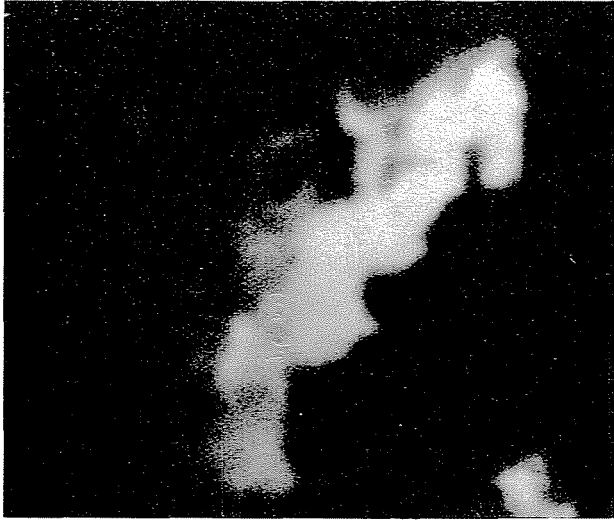


Photo 1

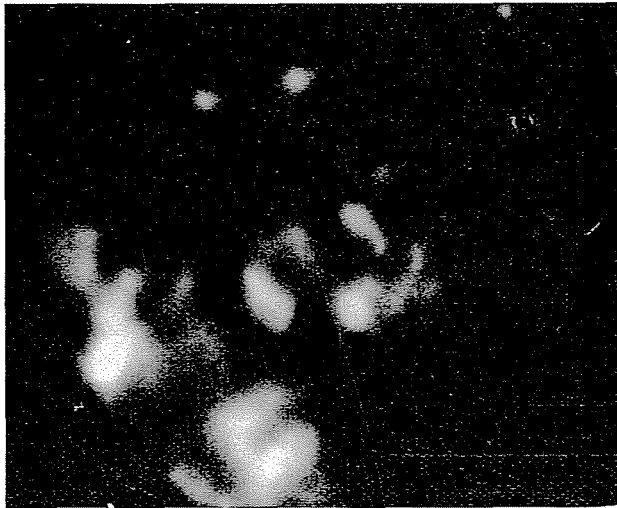


Photo 2

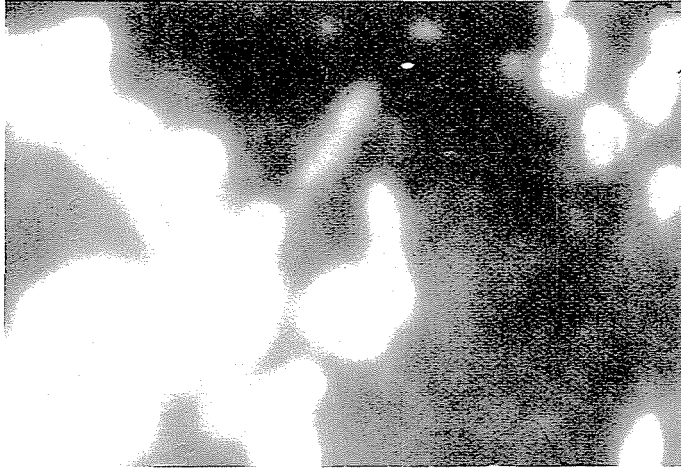


Photo 3

Photos 1—3. Autoradiograms. The distribution of vaseline, labelled with the isotope ^{65}Zn , on the metal surface after 2—3 minutes of degreasing in an alkaline solution

period of 2—3 minutes. On the basis of these autoradiograms, the rate equations discussed above can be explained by the assumption that the high emulsifying action of the bath is not influenced by the metal surface in case of thick grease layers, while in the case of thin grease layers emulsifying power is decreased, and sometimes even practically compensated by adsorptive forces acting between the greases and the metal surface. It is reasonable to assume that in thick grease layers molecules, already present initially in the dimeric form, are emulsified, while molecules in direct contact with the surface have also to be dimerized during their emulsification.

The required purity of the surface

For various anticorrosive coatings that maximum permissible residual quantity of grease impurity on the surface was determined, which does not detrimentally influence the adherence, that is to say, the protective value of the coatings. It was established that degreasing before the application of the lacquer coat has to be carried out to such a degree, so that a grease layer of maximum $5 \mu\text{g}/\text{cm}^2$ shall remain on the surface. This quantity of grease still does not influence the adherence of the lacquer coat. In electroplating, maximum permissible residual grease is $1 \mu\text{g}/\text{cm}^2$. Therefore, before electrodeposition also electrolytic degreasing must always be applied.

Summary

A method, using radioactive tracer technique, was developed for the accurate determination of grease impurities on metal surfaces. This method permits the evaluation of the suitability and the efficiency of various degreasing methods. Greases may be labelled by the iodization of unsaturated oils with the isotope ^{131}I , and with the aid of zinc naphthenate containing isotope ^{65}Zn . From the point of view of measuring techniques, this method is more advantageous than grease indication with isotope ^{14}C .

Experiments showed that the rate and the efficiency of degreasing is practically independent of the chemical composition of the grease impurities.

The rate of degreasing follows an exponential curve, and the process practically stops at a thickness of $2-5 \mu\text{g}/\text{cm}^2$. For the decreasing of this grease layer, electrolytic degreasing can be applied with good results. With this method, the quantity of grease on the surface can be reduced to $1-0.5 \mu\text{g}/\text{cm}^2$. The efficiency of alkaline degreasing baths and the rate of degreasing can be increased by increasing the temperature and the pH-value. In baths of 95°C and of a pH-value above 13, current in industrial practice, the superficial grease layer can be reduced within 15 to 20 minutes to $2-5 \mu\text{g}/\text{cm}^2$, however, at this value degreasing is terminated.

From the point of view of efficiency and rate of degreasing, the role of the surface tension of the bath is unimportant.

Investigations showed that grease impurities on the surface have to be reduced to $2-5 \mu\text{g}/\text{cm}^2$ before lacquer-coating, and to $1 \mu\text{g}/\text{cm}^2$ before electrodeposition, to ensure the good adherence of the coatings.

Literature

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