ELECTROMETRIC METHODS OF ANALYSES APPLIED TO PROCESS AND PRODUCT
CONTROL AT THE AB BOFORS NITROGLYCERINE PLANT.

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ABSTRACT

   Spent acid from the injector process for the production of nitro-
glycerine contains 2 to 3 per cent of nitroglycerine. This may later
separate from the acid. As nitroglycerine is denser than the spent
acid and as part of the acid is recirculated in the production
process careful analysis of nitroglycerine content is required along
with analyses of the contents of nitric and sulphuric acid.

   The nitroglycerine content in the nitrating and spent acids is
determined polarographically in an aqueous solution containing 3
per cent ammonium acetate. The total nitrate content, $\underline{i.\ e.}$ the sum
of nitric acid and nitroglycerine, is determined by titration with
Fe(II) in concentrated sulphuric acid. The sum of nitric and sul-
phuric acid is determined by titration with sodium hydroxide. The
total time for the analyses is about 20 minutes. All analyses are
carried out by instrumental methods. With the aid of a micro
processor controlled titrator the compositions of the acids are
evaluated automatically.

   The nitroglycerine process is controlled by measurement of the
redox potential of the spent acid. The purity of the product nitro-
glycerine is determined by titration with Fe(II) in concentrated
sulphuric acid.

   Results and experiences from the above mentioned methods of
process and product control at the AB Bofors nitroglycerine plant
will be discussed.

   A polagraphic method for the determination of nitrate esters
other than nitroglycerine, $\underline{e.g.}$ mixtures of nitroglycol and nitro-
glycerine, will also be discussed.

INTRODUCTION

   For the purpose of following and controlling the process of

nitration, or rather esterification, of glycerine and similar

alcohols, fast and reliable methods of analysis are required not

just because the final product is highly explosive but also

because of the problems caused by the spent acid.

   In this paper methods of analysis are presented, which are

in use for process and product control in the manufacture of
nitroglycerine by the injector process at AB Bofors.

They are
- polarographic determination of nitroglycerine in nitrating and
  spent acids
- electrometric titration of total amount of nitrate in nitrating
  and spent acids
- potentiometric titration of total amount of strong acid in
  nitrating and spent acids followed by automatic evaluation of
  the composition of the acids
- determination of purity of the nitroglycerine by electrometric
  titration
- on line measurement of the redox potential of the spent acid.

The application of these methods of analysis in the manufacture of
nitroglycerine/nitroglycol mixtures is also discussed.
    Generally, the composition of the acids for nitrations are
chosen such that
- the spent acid from the process can be easily separated from
  the nitrated product
- the solubility of the nitrated product is as low as possible in
  the spent acid
- the spent acid obtained is stable.

    The ratio between the components of the nitrating acid (HNO_3,
H_2SO_4 and H_2O) is important. Sulphuric acid is used as an absor-
bant for water and it is of utmost importance that the content of
sulphuric acid in the nitrating acid is sufficient for efficient
uptake of water formed in the nitration process.
    Nitrating acid for the injector process (composition approxi-
mately 27 % HNO_3, 61 % H_2SO_4, 10 % H_2O and 2 % nitroglycerine;
% stands for mass-%) is obtained by adding oleum and concentrat-
ed nitric acid to the main parts of the spent acid from the process
The rest of the spent acid is sent to denitration. The composition
of the spent acid is approximately 11 % HNO_3, 70 % H_2SO_4, 16,3 %
H_2O and 2,7 % nitroglycerin. This means that the molar ratio H_2O/
H_2SO_4 is about 1.3, i.e. the spent acid contains more water than
does sulphuric acid monohydrate, $H_2SO_4 \cdot H_2O$. The spent acid also
dissolves more nitroglycerine than would an acid with a lower water
content (c.f. figure 1, ref. 1,2) But as the main part of the spent
acid is used in preparing new nitrating acid, losses caused by the
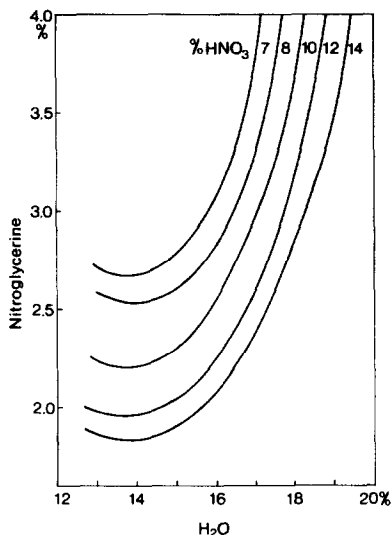
Fig. 1. Solubility of nitroglycerine in spent acid.

increased solubility of the nitroglycerine in the spent acid are
quite small. The solubility of nitroglycerine in the nitrating
acid is larger than in the spent acid and thus there is no danger
of nitroglycerine separating from the prepared nitrating acid.
One should, however, take into account that nitroglycerine may
separate from the spent acid if its composition or temperature
changes unfavourably during storage. To prevent nitroglycerine
from separating from the spent acid sulphuric acid is added.

Nitroglycerine may become highly unstable upon prolonged
contact with acid. V.Öhman et al. (ref. 3) have shown what spent
acid compositions are "safe" and "dangerous" in contact with
nitroglycerine. Their results are shown schematically in figure 2.
The "danger" area is defined as the composition range where heat-
storage of the nitroglycerine phase for 2 hours at 70° C results
in the formation of more than 1 % of nitrous acid (HNO_2). "Normal
safety" corresponds to the formation of 0,2 to 1 % HNO_2, whereas
"high safety" refers to the composition range where less than 0,2 %
HNO_2 is formed - all under the same storage conditions. From the
figure it is evident that the residual acid from the injector
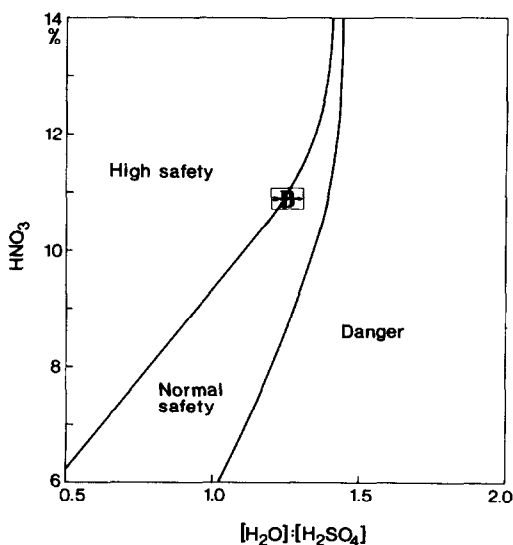process belongs to the area of "high" and "normal" safety.

Fig. 2. Safety diagram of acidic nitroglycerine.

POLAROGRAPHIC DETERMINATION OF NITROGLYCERINE AND NITROGLYCERINE/ NITROGLYCOL IN THE SPENT ACID.

Nitroglycerine.

The nitrate ester group, $-ONO_2$, is reduced irreversibly at a dropping-mercury electrode in a two-electron reaction with formation of the corresponding alcohol and a nitrite ion according to

$$RONO_2 + 2e + H_2O \longrightarrow NO_2^- + ROH + OH^- \tag{1}$$
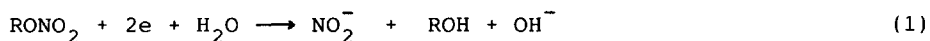
In a water/alcohol solution a well-defined polarographic wave is obtained in the potential range -0.2 V to -1.3 V. Increased alcohol concentration causes the half-wave potential to move towards more negative values. Polarographic reduction of a nitrate ester group in acid solution gives a poorly defined wave. Nitrate ions are also reduced at a dropping-mercury electrode, but at much more negative potentials, about -1.8 V, and thus nitric acid does not interfere in the determination of nitrate esters.

For polynitrate esters the half-wave potential moves towards more positive values with increasing number of nitrate ester groups in the molecule (ref. 4). The change in half-wave potential is ca.
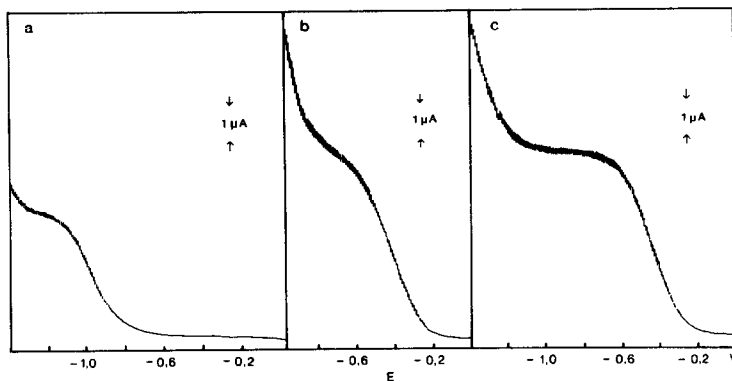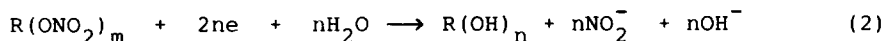
Fig. 3. DC-polarogram for 0.5 mM nitroglycerine i a) water/
ethanol (1:1) containing 3 % NH$_4$Ac, b) water containing 0,03 M
H$_2$SO$_4$ and 0,01 M HNO$_3$, and c) water containing 3 % NH$_4$Ac.

+0.1 V for each new nitrate ester group whereas the diffusion
current increases almost in proportion to the number of added
nitrate ester groups. The overall reaction for reduction of a
polynitrate ester at a dropping-mercury electrode is

$$R(ONO_2)_m \; + \; 2ne \; + \; nH_2O \; \longrightarrow \; R(OH)_n + nNO_2^- \; + \; nOH^- \qquad (2)$$

With DC-polarography only one common polarographic wave over a
relatively large potential range, -0.2 V to -1.5 V, is obtained.

One advantage in polarography is that a linear relationship
between current (signal) and concentration exists over a wide
concentration range.

DC-polarograms are presented in figure 3 for nitroglycerine in
three different solutions, namely a) water/ethanol (1:1) containing
3% NH$_4$Ac, b) water/ethanol 0,03 M H$_2$SO$_4$ and 0.01 M HNO$_3$, and c)
water containing 3% NH$_4$Ac. The figure tells that a 3 % NH$_4$Ac aqueous
solution is a suitable solvent for a polarographic determination of
nitroglycerine.

The concentration of nitroglycerine in the spent acid is relative
ly constant and is about 2.5 to 2.7 mass-%. The nitroglycerine con-
tent in the nitrating acid is somewhat lower but also constant; on
the average determined as 2.2 %. The polarographic determination
gives the total nitroglycerine content in the acid.
Apparatus: Metrohm Polarocord E 506
Referens electrode: Ag/AgCl, Metrohm Ea 427

Procedure: 0.5 g of sample is diluted to 100 ml with distilled water containing 3% NH_4Ac. A DC-polarogram from OV to -1.2V is registered and the diffusion current at -0.8V is measured. A reference solution with 2 g of nitroglycerine per 100 g of an acid mixture, containing 70% H_2SO_4, 17 % H_2O and 13 % HNO_3, is analyzed in the same way.

Nitroglycerine/nitroglycol

With DC-polarography only a common polarographic wave is obtained for nitroglycerine and nitroglycol in the same solution and that technique therefore is not suitable for determination of nitro-glycerine and nitroglycol in spent acid. With differential pulse polarography increased selectivity is obtained and polarographically active substances with differences in half-wave potentials of only 50 to 100 mV can be determined by individual peaks. Even when peaks overlap quantitative determination of the individual components is possible. The highest accuracy is obtained when the determination is carried out in a solvent or solvent mixture where the difference in the polarograms for the components is the largest.

The current i at a given potential (usually the peak current i_p at the peak potential E_p) as a function of concentration can be described by the simply equation

$$i = K \quad C \tag{3}$$

where k is a constant depending on e.g. the pulse amplitude (ΔE), the dropp time (t), the diffusion coefficient (D) and the electrode area (A).

When the solution contains two or more polarographically active substances that are reduced within the same potential range equation (3) can be written as

$$i = \sum_i k_i \, c_i \tag{4}$$

The actual analysis of a mixture is carried out as follows. Suppose the differential pulse polarograms for components A and B have the shapes shown in figure 4. The current for polarographic reduction of a mixture of A and B at potential E_1 and E_2, respectively, can be expressed as

$$i_1 = K_{A1}C_A + K_{B1}C_B \tag{5}$$
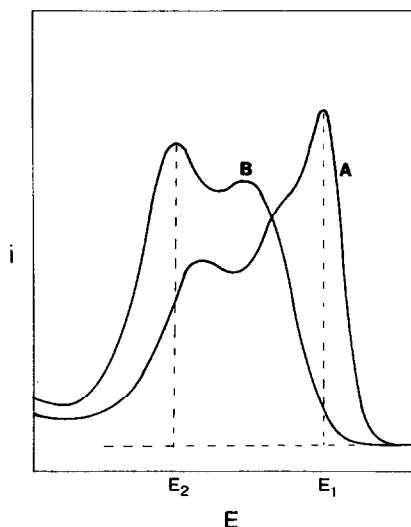
$$i_2 = K_{A2}C_A + K_{B2}C_B \tag{6}$$

Fig. 4. Choice of potentials for differential pulse polarographic analysis of mixtures.

where $K_{A1}, \ldots, K_{B2}$ are constants (or rather the slopes of the calibration curves) for A and B at the potentials E_1 and E_2. With the exception of C_a and C_B, $\underline{i.e.}$ the concentrations of A and B in the sample mixture, all quantities are known or can be measured. From equations (5) and (6) can be derived

$$c_A = \frac{k_{B2} \, i_1 - k_{B1} \, i_2}{k_{A1} \, k_{B2} - k_{A2} \, k_{B1}} \quad \text{and} \tag{7}$$

$$c_B = \frac{k_{A1} \, i_2 - k_{A2} \, i_1}{k_{A1} \, k_{B2} - k_{A2} \, k_{B1}}$$

Values for the constants $k_{A1}, \ldots, K_{B2}$ are determined from standard solutions of pure A and B respectively.

   In figures 5 and 6 differential pulse polarograms are given for nitroglycerine and nitroglycol in water/methanol solutions with ammonium acetate as electrolyte. From the figures can be seen that a sufficiently large difference in the differential pulse polarograms is obtained in a 20 % methanol solution. At this methanol concentration the value of k_1 for nitroglycol has been determined as 0,81 μA mM^{-1} and the value of k_1 for nitroglycerine as 11.38
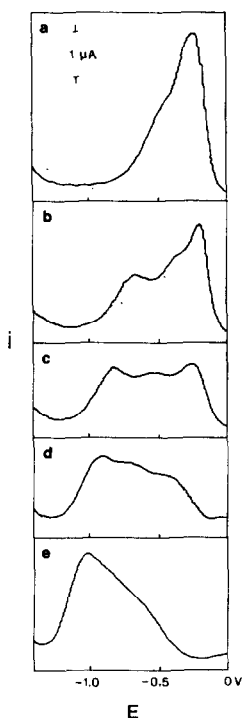
Fig. 5. Differential pulse polarogram for 0.2 mM nitroglycerine in water/ metanol solution, a = 0 %, B = 20 %, c = 40 %, d = 60 % and e = 80 %.
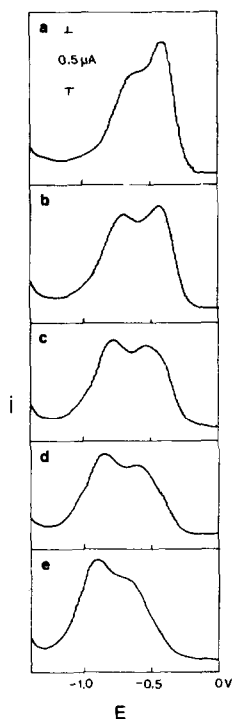
Fig. 6. Differential pulse polarogram for 0.1 mM nitroglycol in water/ metanol solutions, a = 0 %, B = 10 %, c = 20 %, d = 30 % and e = 40 %.

μA mM^{-1} at a potential of -0,24V. The values of k_2 were determined as 7,36 μA mM^{-1} and 5.34 μA mM^{-1}, respectively, at the potential - 0.784V. Figures 5 and 6 show that there are three distinct but overlapping peaks for nitroglycerine where as nitroglycol has two.

Mixtures of nitroglycerine and nitroglycol with well known concentrations have been analyzed. From the results listed in table 1 can be deduced that the mixtures can be analyzed quite accurately. In figure 7 a differential pulse polarogram is shown for a 20 % methanol solution with about equal concentration of nitroglycerine and nitroglycol. In the polarogram there are three distinct but overlapping peaks. The first peak, at ca. -0.25V, is caused almost entirely by the nitroglycerine,

Table 1. Differential pulse polarographic determination of mixtures of nitroglycerine and nitroglycol.

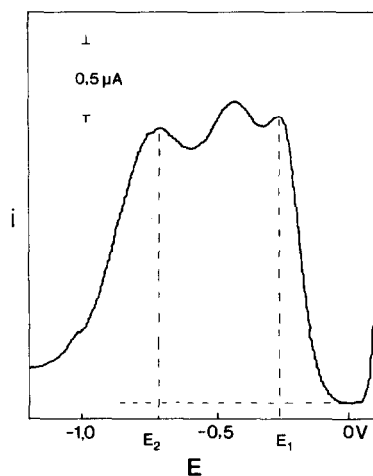| Nitroglycerine, mM		Nitroglycol, mM	
Theoretical	Found	Theoretical	Found
0,1373	0,1377	0.0665	0,0675
0,1355	0,1325	0,0656	0,0664
0,1295	0,1317	0,0627	0,0633
0,1373	0,1373	0,0665	0,0672
0,1413	0,1466	0,1208	0,1226
0,1456	0,1489	0,1245	0,1255
0,1458	0,1478	0,1247	0,1248
0,1440	0,1402	0,1232	0,1227
0,1458	0,1454	0,1924	0,1911
0,1424	0,1429	0,1881	0,1854
0,1523	0,1543	0,2011	0,1988
0,1437	0,1421	0,1898	0,1860

Fig. 7. Differential pulse polarogram for a 20 % methanol solution containing 0,15 mM nitroglycol and 0.175 mM nitroglycerine.

whereas the other two stem from both nitroglycerine and nitro-glycol.

ELECTROMETRIC TITRATION OF TOTAL NITRATE CONTENT IN NITRATING AND
SPENT ACIDS

Standard Fe(II) solutions are often used in analyses of oxidiz-
able substances. In many cases the reactions are fast enough to
allow a direct end-point titration using some suitable redox
indicator or instrumental registration of the course of the
titration.

Depending on the conditions for the reaction nitrate ions react
with Fe(II) ions either in the ratio 1:3 or 1:2.

In hot concentrated hydrochloric acid the following reaction
takes place

$$NO_3^- + 3Fe^{2+} + 4H^+ \longrightarrow NO + 3Fe^{3+} + 2H_2O \qquad (9)$$
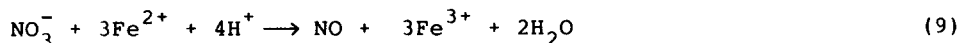
This reaction is too slow to be used for direct titrations.
In the practical applications excess Fe(II) is added and the
reaction is driven to completion by boiling the solution. Excess
Fe(II) is back titrated with Cr(III).

In concentrated sulphuric acid nitrate ions are reduced to
nitrosylsulphuric acid

$$NO_3^- + 2Fe^{2+} + 3H^+ + H_2SO_4 \longrightarrow HSO_3NO_2 + 2Fe^{3+} + 2H_2O \qquad (10)$$
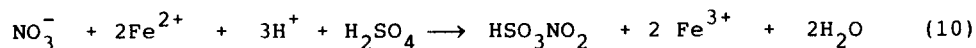
This reaction is sufficiently rapid to allow direct titration
and determinations of total nitrate content in nitrating and spent
acids are based on this reaction.

Nitroglycerine reacts with concentrated sulphuric acid to form
glycerinetrisulphuric acid ester and nitric acid according to

$$C_3H_5(ONO_2)_3 + 3H_2SO_4 \longrightarrow C_3H_5(OSO_2OH)_3 + 3HNO_3 \qquad (11)$$
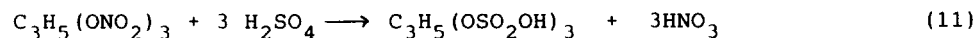
Nitroglycol reacts correspondingly.

The end-point of the titration can be indicated either by the
red complex formed between nitrosylsulphuric acid and the first
excess of Fe(II) ions or instrumentally with two polarized
indicator electrodes (ref. 5). In the instrumental method a
potential is applied across two identical indicator electrodes,
usually platinum, that are immersed in the stirred solution and
the current obtained during titration is measured. Depending on
the reaction components the current tends towards zero at the
equivalence point, goes via a clear minimum or increases from
zero to finite measurable values. By titration of nitrate with
Fe(II) the current goes from zero to a measurable value at the
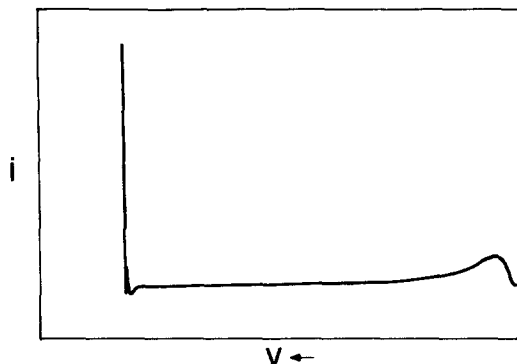equivalence point, since the reduction of nitrate is irreversible

Fig. 8. Current as function of the volume of added Fe(II) for
an electrometric titration of nitrate with two polarized
indicator electrodes.

and the Fe^{2+}/Fe^{3+} system represents a reversibel redox system. The
sudden change in current in the vicinity of the equivalence point
of the titration and facilitates automatic termination of the
titration (c.f. figure 8).

   For the reaction to proceed stoichiometrically according to
equation 10 the temperature in the titration vessel must not
rise above 40 ^{o}C. The water concentration should also be the
lowest possible, something that is achieved by keeping the amount
of sample small.

Apparatus: Metrohm Potentiograph E 536, Multi-Dosimat E 655 and
Polarizer E 585 (polarization potential + 100 mV).

Electrode: Metrohm, double platinum electrode EA 240.

Fe(II) solution: 157 g of ammonium iron (II) sulphate, $(NH_4)_2$
$Fe(SO_4)_2 \cdot 6H_2O$, are dissolved in 700 mL distilled water. The
volume is adjusted to 1000 mL with concentrated sulphuric acid.
The Fe(II) solution is protected against oxygen by a nitrogen
atmoshpere or by a layer of parafin on top of the solution. The
solution is standardized with 200 mg of dried potassium nitrate
(KNO_3).

Procedure: Ca. 600 mg spent acid or ca. 400 mg nitrating acid are
carefully weighed into a beaker containing 20 mL concentrated
sulphuric acid. The sample is allowed to dissolve and mix under
stirring for about 5 minutes. The solution is then transferred to
a double-walled titration vessel. The beaker is rinsed with about
20 mL concentrated sulphuric acid. The double-walled titration

vessel is cooled by + 10 ^{O}C water during the titration. The course
of the titration is registered graphically but the volume of Fe(II)
consumed at the end-point is read from a burett with an electronic
digital display.

POTENTIOMETRIC TITRATION OF THE TOTAL CONTENT OF STRONG ACIDS IN
NITRATING AND SPENT ACIDS AND DETERMINATION OF THE COMPOSITION OF
THE ACIDS.

The total amount of strong acids, sulphuric and nitric acid,
can of course be determined quite easily by visual titration. Here,
however, the titrations have been carried out potentiometrically
with a Metrohm Titroprocessor E 636. With this instrument a complete
evaluation of the composition of the acids are made after input of
the results from the determinations of total nitrate content and
nitroglycerine content. The acid sample to be titrated is weighed
on an analytical balance (Sartorius type 1201 MP 1) directly
linked to the titroprocessor. Only the last part of the titration
curve is registered (figure 9). The results are reported (and
printed) as mass-% HNO_3 (R2), mass-% H_2SO_4 (R3), mass-% H_2O (R4)

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         1.00ML/DIV  V(START)/ML   20.000       PH

      0    1    2    3    4    5    6    7    8    9   10   11   12   13   14
      I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I  I
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       #      1   PH(INIT)    1.392   SMPL.SIZE   0.1660             ROUTINE #1
       1   V/ML  25.727  PH    7.091

       R1   79.4276
       R2   11.3090
       R3   70.6269
       R4   15.2641

       C1 .832500       C3 2.80000      C4 13.6400      C5 .512500
       C6 .778200       C7 100.000
                                        DATE    83.08.24   NAME
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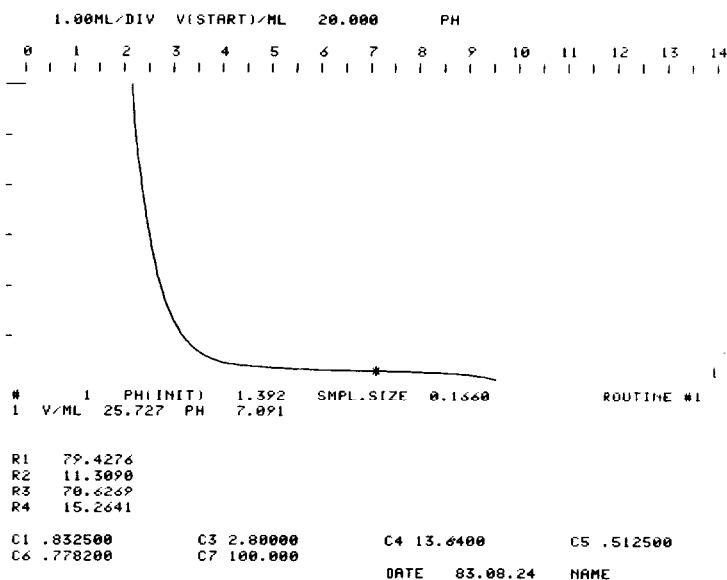
Fig. 9. Potentiometric titration of the total content of
strong acids in spent acid and evaluation of the composition
of the spent acid, R2 = % HNO_3, R3 = % H_2SO_4, R4 = % H_2O and
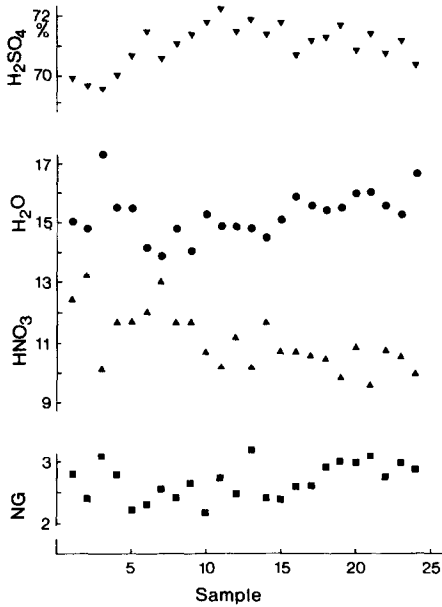C3 = % nitroglycerine.

Fig. 10. Results form determinations of the composition of a
number of spents acids (NG = nitroglycerine).

and mass-% nitroglycerine (C3). The time for a complete analysis
of the composition of a nitrating or spent acid is about 20
minutes. The content of nitrosylsulphuric acid is not determined.

In figure 10 the results form determinations of the composition
of a number of spent acids have been summarized. From the results
it is evident that there are small variations in the composition
of the individual spent acids. These variations must be considered
to be normal. At, relatively speaking, high water concentration
and low nitric acid content a higher value for the amount of nitro-
glycerine is obtained. This is in accordance with the solubility
of nitroglycerine in spent acid as a function of nitric acid and
water content as presented in figure 1. It can also be concluded,
and that is important, that the spent acid samples contained only
dissolved nitroglycerine.

DETERMINATION OF THE PURITY OF NITROGLYCERINE BY ELECTROMETRIC
TITRATION.

The purity of the nitroglycerine is determined by the same
method as that utilized in determinations of total nitrate content

in nitrating and spent acids. In these determinations ca 100 mg,
carefully weighed, of nitroglycerine are used. The time required
for dissolving the nitroglycerine is about 15 minutes, i.e. the
time required for reaction 11 to go to completion. The mean value
for the nitroglycerine content of 50 nitroglycerine batches was
determined as 99.52 mass-% with a standard deviation of 0.19 mass-
%.

ON-LINE MEASUREMENT OF THE REDOX POTENTIAL OF THE SPENT ACID.
    Measurement of the redox potential in nitric acid and in mix-
tures of nitric acid and nitrogen oxides have been made for a
long time. V. Öhman et al. (ref. 3,6,7) studied the redox potential
of acid mixtures in connection with studies of the stability of
spent acids from nitroglycerine and nitroglycol manufacturing
processes. They also tried to explain the electrode processes.
    The redox potential in the system $HNO_3-HNO_2-H_2O$ with nitric
acid concentrations up to 12 mol/L (56 mass-%) is determined by
the following reaction

$$HNO_3 + 2H^+ + 2e \rightarrow HNO_2 + H_2O \tag{12}$$

The expression below is valid for the redox potential at 25 $^{\circ}$C when
the content of nitrous acid is small compared to that of nitric
acid and when the nitric acid and hydrogen ion activities can be
regarded as constant

$$E = E_o + 0.0296 \log \frac{\{HNO_3\}\{H^+\}^2}{\{HNO_2\}\{H_2O\}} \tag{13}$$

were E_o is the standard potential at equilibrium.
    Equation (13) is difficult to apply to the $HNO_2-HNO_3-H_2SO_4-$
H_2O systems that are of interest in the production of nitrate
esters because of the complex dissociation equilibria in these
systems. V. Öhman (7) have shown that NO_2^+ and NO^+ ions influence
the redox potential at low water concentrations. They also showed
that a linear logarithmic relationship exists and that a break
point is obtained for sulphuric acid monohydrate $H_2SO_4 \cdot H_2O$.
Dissolved nitroglycerine does not affect the redox potential
very much. There is however, a considerable effect when decompo-
sition of the spent acid occurs.
    At the AB Bofors nitroglycerine plant for more than a year now
continuous measurment of the redox potential of the spent acid
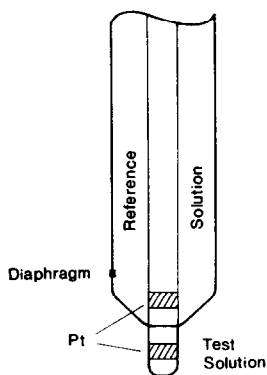
Fig. 11. Measuring cell for determination of the redox potential.

have been made immediately after the separator as a complement
to the laboratory determinations of the composition of the acid.
An Ingold Pt-488 electrode is used as the measuring cell. It is
used in combination with another platinum electrode and the set-up,
in principle, functions as a concentration cell (figure 11). As
reference solution an acid mixture corresponding to spent acid is
used. It does not, however, contain any nitroglycerine.

To examine the response of the electrode to changes in compo-
sition of the acid mixture the redox potential was measured in
H_2SO_4-H_2O, HNO_3-H_2O and H_2SO_4-HNO_3-H_2O mixtures with varying
water content. The results are summarized in figure 12. They are
given in terms of the change in redox potential, ΔE, as a function
of the logarithm of the molality of water in the mixtures, log m.
The potential change at the beginning of each experiment was taken
as zero. The absolute potential for the different mixtures varies
and is a function also of the composition of the reference solution.
The figure shows that the change in redox potential for the HNO_3-
H_2O system is negative in comparison with the change in redox
potential for both the H_2SO_4-H_2O and the H_2SO_4-HNO_3-H_2O systems.
It is also of interest to note that the difference in potential
change for the H_2SO_4-H_2O and the HNO_3-H_2O systems corresponds
approximately to the change in redox potential for the H_2SO_4-
HNO_3-H_2O system.

At very low water contents changes in redox potential are
small, but at higher water concentrations the changes are larger
and a linear relationship is found between the change in redox
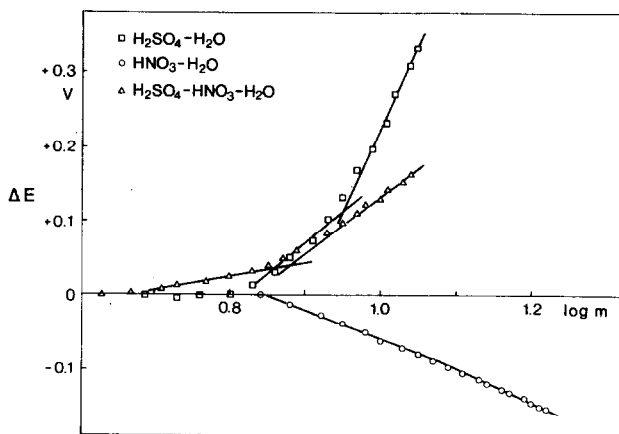potential and the logarithm of the molality of water, with clear

Fig. 12. The change in redox potential, ΔE, as a function of the logarithm of the molality of water in the mixtures, log m.

break points at the molar ratio $H_2SO_4:H_2O = 1$ in the $H_2SO_4-H_2O$ (log m = 0,95) and the $H_2SO_4-HNO_3-H_2O$ (logm = 0,89) systems. For the HNO_3-H_2O system a break point is only just detectable for the molar ratio $HNO_3:H_2O = 1$ (log m = 1.09). The spent acid from the nitroglycerine process has a water content corresponding to a value of log m of ca. 0.92. Figure 12 shows that ΔE as a function of log m is linear in that water content range.

The redox potential of the spent acid is measured continuously during the nitroglycerine manufacture. Under normal processing conditions a stable signal is obtained, but under deviating conditions, e.g. when the spent acid contains too much water, this shows up as a change in the redox potential. The experience from measurment of the redox potential are thus extremely good. The personnel are now able to control the process, i.e. increase or decrease the flow of glycerine, not only with the aid of tempera-ture measurements but also by observing changes in redox potential in the spent acid. This also seems to give the operator an enhanced "feeling" for the process.

DISCUSSION

Electrometric methods for analysis have turned out to be very
suitable for controls of the nitroglycerine process. Polarography
can be used not only for the determination of nitroglycerine in
the spent acid but also for determination of nitrate ester content
in the water from the soda wash. In that water glycerine is
present both as di- and trinitrate, both of which can be analyzed
for polarographically as described for two-component systems in
2.2. With polarography it is also possible to follow the hydroly-
sis of nitroglycerine in alkaline solution.

REFERENCES

1 G. Wallerius, Ingeniörsvetenskapsakademiens Handlingar Nr 113,
   Stockholm, 1931, 52 pp.
2 H.J. Klassen and J.M. Hamphrys, Chem. Eng. Progr., 49 (1953),
   641-646.
3 V. Öhman, E. Camera and L. Cotti, Explosivstoffe, 6 (1960),
   120-127; 7 (1960), 148-158.
4 G.C. Whitnack, J.M. Nielsen and E.St.C. Gantz, J.Am.Chem.Soc.,
   76 (1954) 4711-4714.
5 A.F. Williams and J. Brooks, Proceedings of the International
   Symposium on Microchemistry, 1958, pp 430-438.
6 V. Öhman, Chemiker-Ztg., 82 (1958), 651-656.
7 V. Öhman, Svensk Kemisk Tidskrift, 78 (1966), 20-34.