VOC-removal by means of non-thermal plasmas: plasma chemistry, techniques and examples

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3rd International PlasTEP Summer School

Kaunas, July 2012
VOC reduction by non-thermal plasmas

Content

1. Introduction
   - VOCs and state of the art removal

2. VOC-removal by means of plasmas
   - Plasma generation in gas discharges
   - Plasma chemistry for VOC-removal
   - Plasma enhanced / hybrid processes
   - Examples for VOC-removal and benefits of the technology

3. Summary
1. Introduction
Volatile Organic Compounds (VOCs)

“Definition”
- organic chemical compounds with high enough vapour pressures
  (normal conditions) to significantly vaporize and enter atmosphere

- most common methane (CH\textsubscript{4})
  $\rightarrow$ greenhouse gas
- “non-methane VOC” NMVOC:
  wide range of carbon-based molecules;

  e.g. aldehydes, ketones, aromatic and light hydrocarbons (xylol, toluol),
  alcohols, acetates, bencines, glykoles, formaldehyde
1. Introduction
VOC sources

Natural
- natural gas
- trees, wetlands, ...

Artificial:
- agriculture, burning biomass
- industry: used paint thinners, dry cleaning solvents, constituents of fuels (e.g. gasoline, natural gas), brownfield sites
- transportation: crude oil tanking
- “civilisation”: photocopiers, carpets, furnishings, office components, wall covers, paints (sick building syndrome), tobacco smoke
1. Introduction
NMVOC emission sources

Emissionen flüchtiger organischer Verbindungen ohne Methan (NMVOC) nach Quellkategorien

Tsd. t

3 759
3 500
3 300
3 100
2 900
2 700
2 500
2 300
2 100
1 900
1 700
1 500
1 300
1 100
900
700
500
300
100
0


Verkehr, ohne land- und forstwirtschaftlichen Verkehr
Haushalte und Kleinverbraucher, mit land- und forstwirtschaftlichem Verkehr sowie Militär

Energy industry (combustion) Manufacturing industry Transport
Residential and comercial comb. Fugitive Emissions from Fuels Industrial processes (chemistry)
Solvent and other product use Agriculture


R. Brandenburg, "VOCs", 3rd Summerschool
### 1. Introduction

#### Typical VOCs and their health effect

<table>
<thead>
<tr>
<th>VOCs</th>
<th>TLV (ppm)</th>
<th>Chemical Symbols</th>
<th>Effects on Humans</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone</td>
<td>10</td>
<td>CH₃COCH₃</td>
<td>Carcinogen</td>
</tr>
<tr>
<td>Benzene</td>
<td></td>
<td>C₆H₅</td>
<td>Carcinogen</td>
</tr>
<tr>
<td>Butyl Acetate</td>
<td></td>
<td>CH₃COOC₄H₉</td>
<td>Cacinogen</td>
</tr>
<tr>
<td>Carbon Tetrachloride</td>
<td>10</td>
<td>CCl₄</td>
<td>Probable carcinogen</td>
</tr>
<tr>
<td>Dichloroethane</td>
<td></td>
<td>CH₂ClCH₂Cl</td>
<td>Paralysis of nerve centres</td>
</tr>
<tr>
<td>EGM</td>
<td>5</td>
<td>(CH₃OH)₂(C₂H₅)₂C</td>
<td>Birth defects</td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td></td>
<td>C₆H₅C₂H₅</td>
<td>Probable carcinogen</td>
</tr>
<tr>
<td>Formaldehyde</td>
<td></td>
<td>HCHO</td>
<td>Sore throat dizziness and headache</td>
</tr>
<tr>
<td>Methyl chloride</td>
<td></td>
<td>CH₃Cl</td>
<td>Carcinogen</td>
</tr>
<tr>
<td>Methyl Ethyl Ketone</td>
<td></td>
<td>CH₃COC₂H₅</td>
<td>Carcinogen</td>
</tr>
<tr>
<td>Perchloroethylene</td>
<td></td>
<td>C₂H₅Cl</td>
<td>Probable carcinogen</td>
</tr>
<tr>
<td>Phenol</td>
<td></td>
<td>C₆H₅OH</td>
<td>Headache, dizziness</td>
</tr>
<tr>
<td>Styrene</td>
<td></td>
<td>C₆H₅CH=CH₂</td>
<td>Probable carcinogen</td>
</tr>
<tr>
<td>Tetrachloroethylene</td>
<td></td>
<td>CCl₂=CCl₂</td>
<td>Probable heart and liver disease</td>
</tr>
<tr>
<td>Toluene</td>
<td></td>
<td>C₆H₅CH₃</td>
<td>Headache, dizziness</td>
</tr>
<tr>
<td>Trichloroethane</td>
<td>200</td>
<td>CH₂ClCHCl₂</td>
<td>Probable birth defects, heart problems</td>
</tr>
<tr>
<td>Trichloroethylene</td>
<td>50</td>
<td>CHCl=CCl₂</td>
<td>Liver and kidney disease, paralysis of nerve centres</td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>10</td>
<td>CH₂CHCl</td>
<td>Carcinogen</td>
</tr>
<tr>
<td>Xylene</td>
<td></td>
<td>C₆H₆(CH₃)₂</td>
<td>Headache, dizziness</td>
</tr>
</tbody>
</table>

### TLV – threshold limit value
1. Introduction

VOC-decomposition and deodorization methods

**Thermal Processes**
1. TO, Thermal Oxidation
2. RTO, Regenerative Thermal Oxidation
3. Catalytic Oxidation with Recuperation

**Filtering/Adsorption**
4. Biofilters
5. Scrubber
6. Adsorption Container
7. Concentrator Unit with TO
8. Filtering

**Non-thermal Oxidation**
6a. Electrical Non-thermal oxidation
6b. UVS Non-thermal Oxidation

Haus der Technik, Essen/GER
Depollution of gases with NTP-Technology

Plasma assisted processes
- Electron Beam Flue Gas Treatment
- Ozone Injection (LoTOx)
- Wet Scrubbing Pre-Treatment (ECO)

\[ \text{NO oxidation to } \text{NO}_2, \text{N}_2\text{O}_4, \text{N}_2\text{O}_5 \text{ and reduction with } \text{NH}_3 \]

Plasma based processes
- Electrostatic Precipitators (mechnical → dust filtration)
- Deodorization
- Non-thermal VOC removal

Plasma based incineration
- Thermal destruction of waste and haszardous gases by (hot) plasma torches

R. Brandenburg, "VOCs", 3rd Summerschool
VOC reduction by non-thermal plasmas

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   - Benefits of the technology

3. Summary
Plasma based exhaust treatment

**Barrier discharge**

- Gas
- Dielectric electrode
- Grounded electrode

**Corona discharge**

- High voltage
- Gas
- Wire electrode
- Grounded electrode

**Ozoniser; Deodorization**

**Electrostatic precipitators**

R. Brandenburg, "VOCs", 3rd Summerschool
2. VOC decomposition in non-thermal plasmas

**Dielectric barrier discharge (DBD)**

**Volume DBD**

**Surface DBD**
2. VOC decomposition in non-thermal plasmas

**Stack reactor DBD**

*Stack system with structured electrodes*

S. Müller, R.-J. Zahn; Contributions to Plasma Physics 47 (2007) 520-529
Mobile NTP-Reactor for field tests

- Up to 1.000 Nm³/h
- Combination with different Adsorbents/Catalysts
- Mobile Analytics incl.
Particle filtration: Ion wind

Extraction of ions from surface DBD
- Charging and electrostatic precipitation of particles
- Additional effects on gases and deposited organic particles due to radicals and oxidation

2. VOC decomposition in non-thermal plasmas Surface DBD with ion-extraction

S. Müller, R.-J. Zahn, J. Grundmann; Plasmas and Polymers 4 (2007) S1004
Special types of DBD and corona

Packed Bed Reactor

Corona Radical Shower

J.S. Chang et al, McMaster Uni/Can
2. VOC decomposition in non-thermal plasmas (Corona) radical shower

- Applied in particular to NOx-removal
- Plasma treats only a portion of gas flow, creating active species, which then treat the total gas flow as a „shower“

J.S. Chang et al, McMaster Uni/Can
### 2. VOC decomposition in non-thermal plasmas

### Atmospheric pressure plasmas

#### Non-Thermal (NT) Plasmas

**“Cold” Non-Thermal Plasmas**

- \( T_i \approx T_g \approx 300 \ldots 400 \text{ K} \)
- \( T_i \ll T_e < 10^5 \text{ K} \) (10 eV)

#### Translational (“Hot NT”) Plasmas

- \( T_i \ll T_e \leq 10^4 \ldots 10^5 \text{ K} \)
- \( T_i \approx T_g \leq 4 \times 10^3 \text{ K} \)

#### Thermal Plasmas

**Thermal Plasmas**

- \( T_i \approx T_g \approx T_e \)
- \( T_x < 5 \times 10^3 \ldots 10^4 \text{ K} \)

#### Types of Non-Thermal Plasmas

1. **Barrier discharges**
2. **Coronas**
3. **Microplasma-Arrays**
4. **Plasma jets**
5. **Gliding Arc**
6. **Plasma Torch**
7. **Arc**
8. **Arc jet**

#### Microwave Driven Plasmas
Plasma at work

**Volume DBD in atmospheric air**

- **Electrode**
- **Dielektric**

**Microdischarges**
- Electrical breakdown
- Initiation of chemical processes

R. Brandenburg, "VOCs", 3rd Summerschool
Overall process of plasma based removal

1. Breakdown phase (ps ... ns)
   - Ionisation, Dissociation, Excitation ... → Ions, Electrons & Radicals

2. Reaction phase (µs ... ms)
   - Recombination and conversion of ions and radicals
     (primary radicals OH, O → secondary radicals O₃, HO₂, ...)
   - Oxidation of pollutants
   - Surface reactions (activation, structural changes)

3. Post phase (ms ... s)
   - Diffusion, transport of heat and material, chemical reactions with post reactants
   - Aerosol formation and adsorption
VOC-removal in plasma

Free electrons: $e^- + \{O_2, H_2O, \ldots\} \rightarrow OH, M^+, M^- \rightarrow HO_2, O_3, \ldots$

$\Rightarrow$ Saturated Hydrocarbons:

Dehydrogenization

$\begin{align*}
R-H + O & \rightarrow R + OH \\
R-H + OH & \rightarrow R + H_2O & R\cdot \ldots \text{ organic radical}
\end{align*}$

Oxidation

$\begin{align*}
R + O_2 & \rightarrow R-O-O & R-O-O \ldots \text{peroxy radical}
\end{align*}$

$\text{Further oxidation to } CO_2 \text{ and } H_2O$

$\text{Radical chain reaction}$

$R_a-O-O + R_b-H \rightarrow R_aOOH + R_b$

$ROOH \ldots \text{alkyl hydroperoxide}$

$\Rightarrow$ Unsaturated Hydrocarbons:

Additionally radical addition following oxidation, radical chain reaction or polymerisation of hydrocarbons
2. VOC-reduction plasma chemistry

Example: Formaldehyde (CH\textsubscript{2}O)

- destruction of CH\textsubscript{2}O results dominantly from chemical attack by OH and O radicals
- primary end products: CO, H\textsubscript{2}O
- destruction rates typically 2-8 ppm/(1 J/l)

2. VOC-reduction plasma chemistry

DMS removal by pulsed negative corona

\[ \text{OH} + \text{CH}_3\text{SCH}_3 \rightarrow \text{CH}_2\text{S} + \text{CH}_3\text{OH} \]
\[ \text{OH} + \text{CH}_3\text{SCH}_3 \rightarrow \text{H}_2\text{O} + \text{CH}_2\text{SCH}_2 \]

\[ \text{CH}_3\text{SCH}_2 + \text{O}_2 + \text{M} \rightarrow \text{CH}_3\text{SCH}_2\text{OO} + \text{M} \]
\[ \text{CH}_3\text{SCH}_2\text{OO} + \text{NO} \rightarrow \text{CH}_3\text{SCH}_2\text{O} + \text{NO}_2 \]
\[ \text{CH}_3\text{SCH}_2\text{O} + \text{M} \rightarrow \text{CH}_3\text{S} + \text{CH}_2\text{O} + \text{M} \]

\( \text{CH}_3\text{S} \rightarrow \text{Oxidation to CO}_2, \text{H}_2\text{O}, \text{SO}_2 \)

\[ \text{OH} + \text{CH}_3\text{SCH}_3(+\text{M}) \leftrightarrow \text{OH} \cdot \text{CH}_3\text{SCH}_3(+\text{M}) \]
\[ \text{OH} \cdot \text{CH}_3\text{SCH}_3 + \text{O}_2 \rightarrow \text{HO}_2 + \text{CH}_3\text{S(O)CH}_3 \]
\[ \text{OH} \cdot \text{CH}_3\text{SCH}_3 + \text{O}_2 \rightarrow \text{OH} + \text{CH}_3\text{S(O)CH}_3 \]

\( \text{CH}_3\text{S(O)CH}_3, \text{CH}_3\text{S(O)CH}_3 \rightarrow \text{Reaction with OH} \)
\( \rightarrow \text{Adsorption in water} \)

Gutsol and Fridman, Drexel University
### Reaction rates

<table>
<thead>
<tr>
<th>Reaction</th>
<th>Two body reaction rate [cm$^3$/s]</th>
<th>Three body reaction rate [cm$^6$/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>molecule – molecule</td>
<td>$10^{-14}$ - $10^{-31}$</td>
<td>$10^{-30}$ - $10^{-40}$</td>
</tr>
<tr>
<td>atom / radical – molecule</td>
<td>$10^{-11}$ - $10^{-24}$</td>
<td>$10^{-30}$ - $10^{-36}$</td>
</tr>
<tr>
<td>ion – atom / molecule</td>
<td>$10^{-9}$ - $10^{-13}$</td>
<td>$10^{-28}$ - $10^{-32}$</td>
</tr>
<tr>
<td>electron – molecule / molecule</td>
<td>$10^{-7}$ - $10^{-11}$</td>
<td>$10^{-27}$ - $10^{-35}$</td>
</tr>
<tr>
<td>positive – negative ion</td>
<td>$10^{-3}$ - $10^{-8}$</td>
<td>$10^{-25}$ - $10^{-26}$</td>
</tr>
<tr>
<td>electron – ion</td>
<td>$10^{-6}$ - $10^{-7}$</td>
<td>$10^{-26}$ - $10^{-26}$</td>
</tr>
<tr>
<td>molecule / radical-aerosol</td>
<td>$(10^{-5}$ - $10^{-10}$) Rp(nm)</td>
<td>Rp: diameter of aerosol</td>
</tr>
</tbody>
</table>

### Comparison of dominant reaction

\[
\text{[reaction rate]} \times \text{[molecule density]} \times \text{[reactant density]} \times \text{[third body molecule density]}
\]
Ionic vs. Radical reactions

\[ A + B \xrightarrow{k_1} N + M \quad \text{A ... radical; N ... product} \]

\[ C^+ + B \xrightarrow{k_2} N + L \quad \text{C\textsuperscript{+} ... ion} \]

Kinetic equation:

\[ \frac{dN}{dt} = k_1[A][B] + k_2[C^+][B] - \cdots \]

However,

\[ [A] \gg [C^+] ; k_1 \ll k_2 \]

Thus,

\[ k_1[A] \approx k_2[C^+] \]

NTP vs. RTO

NTP-VOC removal:
10 – 30 eV/VOC-molecule

Regenerative Thermal Oxidation (RTO):
0.1 eV/molecule per molecule of air

Lower energy consumption in NTP if VOC-concentration > 0.3 ... 1% (3.000 – 10.000 ppm)

Adapted from Fridman, Drexel University
2. VOC-reduction plasma chemistry

Evaluation

- Specific Energy Density (Spec. Energy Input SEI)
  \[ S_{\text{ED}} (J/L) = \frac{P_{\text{dis}}}{Q} \]
  \( P_{\text{dis}} \) ... dissipated plasma power; \( Q \) ... gas flow

- \( \text{CO}_2 \)-Selectivity \( S_{\text{CO}_2} \)

- Carbon balance CB

- Decomposition efficiency \( h \) (Destruction and removal efficiency, DER)

- \(\eta\) (%)
  \[ \eta \% = \frac{[\text{VOC}]_0 - [\text{VOC}]}{[\text{VOC}]_0} \times 100 \]
  \( [\text{VOC}]_0 \) ... inlet concentration; \( n \) ... number of C-atoms

Kim, Plasmas and Polymers 2004
Evaluation: SED-plots

\[ \frac{[X]}{[X]_0} = \exp\left(-\frac{\text{SED}}{\beta}\right) \]

\[ \text{SED} = -\beta \ln\left(\frac{[X]}{[X]_0}\right) \]

\[ 1/\beta = k_E \ldots \text{energy constant} \]

\[ k_E = f(\text{Temp, gas comp., } [VOC]_0, \ldots) \]

Veldhuizen, TU Eindhoven

R. Brandenburg, "VOCs", 3rd Summerschool
Energy constants

TCE in Ar-\(\text{O}_2\)

\[
\begin{array}{c}
\text{Cl} \quad \text{H} \quad \text{Cl} \\
\text{Cl} \quad \text{C} \quad \text{C} \\
\text{H} \quad \text{H}
\end{array}
\]

Trichloroanisol (TCA) in \(\text{N}_2\)

\[
\begin{array}{c}
\text{C}_7\text{H}_5\text{Cl}_3\text{O} \\
\end{array}
\]

Exponential-folding values \(\beta\) for example gas mixtures (straight-line removal plot approximations).

<table>
<thead>
<tr>
<th>Gas mixture</th>
<th>(\beta) value (J/\text{std l})</th>
</tr>
</thead>
<tbody>
<tr>
<td>TCE in dry Ar–(\text{O}_2)</td>
<td>12</td>
</tr>
<tr>
<td>TCE in humid Ar–(\text{O}_2)</td>
<td>95 (at 3 exp. folds)</td>
</tr>
<tr>
<td>(\text{CCl}_4) in dry Ar–(\text{O}_2)</td>
<td>520</td>
</tr>
<tr>
<td>(\text{CCl}_4) in humid Ar–(\text{O}_2)</td>
<td>1824</td>
</tr>
<tr>
<td>TCA in dry (\text{N}_2)</td>
<td>208 (at (\leq 4) exp. folds)</td>
</tr>
<tr>
<td>Toluene in dry (\text{N}_2)</td>
<td>300</td>
</tr>
</tbody>
</table>

Veldhuizen, TU Eindhoven

R. Brandenburg, "VOCs", 3rd Summerschool
Energy cost

- Energy Price significantly depends on initial concentration
- Few ppm: energy price reaches very high values (not all active species can target VOC molecules)
- Higher concentrations: fraction of energy for removing pollutant molecules higher and energy spent for elimination of each single molecule decreases

Gutsol and Fridman, Drexel University
2. VOC-reduction plasma chemistry

Aerosol particle formation

Reactions of larger radicals (CHO, CHN) with cluster ions and molecules

Generation of nitric acid (HNO$_3$) $\rightarrow$ reaction with radicals

Promotion of VOC removal due to heterogeneous reactions

![Graph showing aerosol formation from the decomposition of benzene](image)

Figure 18. Aerosol formation from the decomposition of benzene (200 ppm benzene, 0.5 vol.-% water vapor). Aerosol was measured by SMPS (TSI Co.).

Kim, Plasmas and Polymers 2004

R. Brandenburg, "VOCs", 3rd Summerschool
2. VOC-reduction plasma chemistry

Non-thermal plasma features for VOCs

+ Decomposition of contaminants without heating
+ Wide range of pollutants (Gases ... Particulate Matter PM)
+ Decomposition of organic PM
+ High efficiency for low contamination (e.g. deodorization) 
  \([\text{VOCs}] < 1 \text{ g C}_{\text{org}}/\text{m}^3\)]

- High energy cost/molecule \(\rightarrow\) high energy for high concentrations
- Uncompleted conversion and by-products \(\rightarrow\) low selectivity (\(\text{CO}_2\))
- Deposition of polymer films in reactors \(\rightarrow\) unstable plasma source

Possibilities

- Indirect treatment
- Hybrid methods = combination of plasma with ...
  ...
  ... catalysts
  ... adsorbents
  ... scrubbing

Heterogeneous reactions and synergies!
Plasma and catalysts

Dichlormethane (DCM) vs Benzene

- Packed bed reactors with catalytic filling (TiO$_2$) with best performance
  - Shift of temperature
  - Decrease of required specific energy density
  - Increase of selectivity

Whitehead et al, Manchester Uni.; Kim, AIST Korea

R. Brandenburg, "VOCs", 3rd Summerschool
Plasma & Catalyst

**Plasma**
- Enhancement of retention time and concentration
- Increase of electron temperature and density
- Voltage potential across
- Local heating
- Active species

**Catalyst**
- Adsorption of pollutants
- Electric field enhancement
- Increase of work function
- Surface regeneration
- Increase of surface area
- Change of oxidation stage
- Formation of active sites
- Enhance dispersion of active components

Enhance energy efficiency  Improve selectivity  Extend cat. durability

Synergy between plasma and adsorber

- zeolite adsorber (w/o plasma) and undecane C_{11}H_{26} as model gas

- Removal of undecane by plasma
  → by-products: CO\textsubscript{2} and formic acid (CH\textsubscript{2}O\textsubscript{2} / HCOOH)

- Longer time until breakthrough of adsorber with plasma on
  → plasma treatment increases duration of adsorption

- Synergy effects:
  → Activation of adsorber
  → Removal of adsorbed VOCs
      (“in-situ regeneration”)
  → Load reduction by NTP-removal
  → Removal of plasma-byproducts

- Literature describes cycled processes:
  (1) absorption w/o plasma
  (2) decomposition by O\textsubscript{2}-plasma

R. Brandenburg, "VOCs", 3rd Summerschool
2. VOC decomposition in non-thermal plasmas

**Cycled process**

- **Adsorption (Plasma OFF)**
  - VOCs
  - Adsorbent/Catalyst
  - Clean gas

- **Cycled operation**
  - Oxygen
  - CO$_2$, H$_2$O

- **VOC decomposition by O$_2$ Plasma**

Kim, AIST/JP
2. VOC decomposition in non-thermal plasmas

**Cycled process**

![Diagram of VOC decomposition process](image)

**Figure 8.** VOC removal apparatus. (a) Gas flow system and (b) packed bed plasma reactor.

Mizuno, Toyohashi University
2. VOC decomposition in non-thermal plasmas

Deodorization unit (commercial)

Sunamachi wastewater treatment center (Tokyo)

Plasma-CAT (UTD Co., Swiss)

Two-stage Plasma-Catalyst System

Flow rate = 55,000 Nm³/hr
Odor concentration =
Removal efficiency = 94%~99%
Specific input energy =
Construction cost = ¥ 140,000,000

Kim, AIST Japan
2. VOC decomposition in non-thermal plasmas

Multi-stage treatment with molecular sieves

Catalytic supported NTP plant for 10,000 m³/h of waste air

Figure 11: Catalytic supported NTP plant for 10,000 m³/h of waste air behind flavouring processes for food

1. Enrichment of high-molecular compounds in molecular sieve buffer
2. Oxidation of odours with a plasma stage
3. Regeneration of loaded molecular sieve by catalytic thermal oxidation
2. VOC decomposition in non-thermal plasmas
Bypass operated plasma plants

Figure 14: Diagram of a NTP plant for odour reduction in factories for producing fattening food and fish meal (very humid emissions)

- Indirect plasma treatment of polluted gas by plasma treated gas

R. Rafflenbeul, Envisolve.com; Germany
2. VOC decomposition in non-thermal plasmas

Economical benefit

Investment- and running costs

Figure 15: Investment- and running cost comparison of waste air purification processes (50,000 m³N/h) for <100 mg VOC/m³ in the flavour processing industry

R. Rafflenbeul, Envisolve.com; Germany
Commercial systems for deodorization

- Commercialized NTP system

(Original format from J.A. Dyer, K. Mulholland, Chem. Eng., pp. 4-8, 1994)

Kim, AIST Japan
3. Summary

- VOCs can be decomposed in non-thermal plasmas (NTPs) via „radical based“ plasma chemistry.

- In practice plasmas are combined with other technologies (adsorbents, catalysts) in order to improve efficacy and selectivity.

- Exhaust treatment by means of NTP is especially suited for low concentrations.

- Applicability/feasibility is determined by the specific situation (type and amount of contaminants, properties of gas flow) and has to consider efficacy and selectivity.
“There are more things between anode and cathode than dreamt of in our philosophy!” [sic!]

H. Raether, (1909-1986)