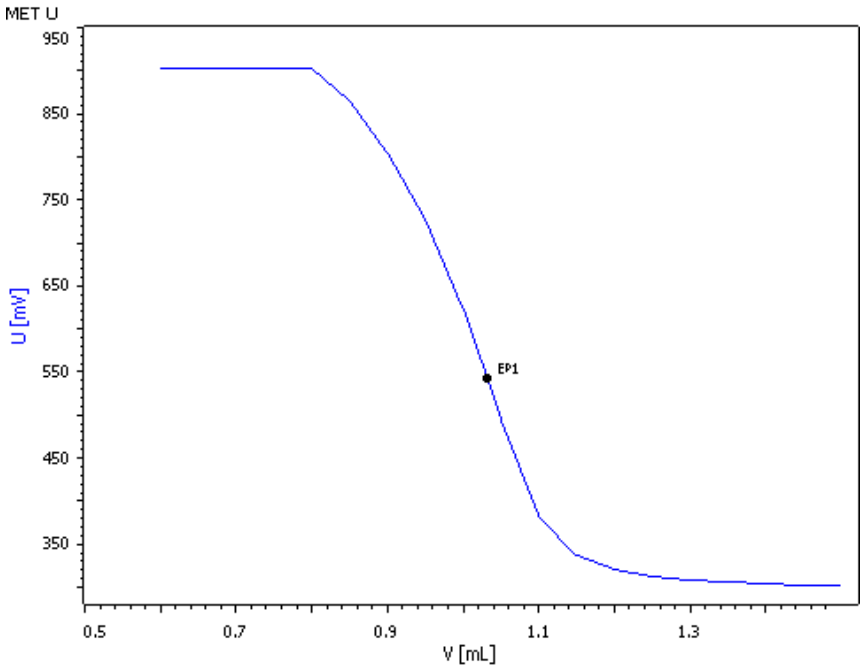
Titration Application Note T-087

# Carboxyl end groups in polymers

## Photometric determination based on ASTM D7409




The carboxyl end groups (CEG) in polymers, such as polyethylene terephthalate (PET), are a measure of the number of unreacted carboxylic acid groups at each end of a polymer chain. The number of CEGs may influence the hydrolysis resistance of geosynthetics, such as geogrids and geotextiles. The lower the CEG value the higher is the hydrolysis resistance of geosynthetics, which in turn increases their stability.

This Application Note describes the photometric titration of carboxyl end groups in PET pellets using the Metrohm Optrode. The acidic end groups of the polymer are titrated with an ethanolic KOH solution using bromophenol blue as indicator.

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# Method description

### Sample

Polyethylene terephthalate (PET) pellets

### Sample preparation

Approximately 1.0–1.6 g PET pellets are weighed into a 50 mL Erlenmeyer flask and 20 mL o-cresol is added. It might be that the sample quantity and the procedure have to be adapted to prevent precipitation. The solution is boiled while stirring under reflux until the sample is completely dissolved (approx. 20 minutes).

The solution is cooled down to room temperature and 35 mL chloroform and 0.04 mL bromophenol blue are added.

### Configuration

| | |
|---|---|
| 905 Titrando | 2.905.0010 |
| 800 Dosino | 2.800.0010 |
| Dosing unit 5 mL | 6.3032.150 |
| 801 Stirrer | 2.801.0040 |
| Optrode (λ = 610 nm) | 6.1115.000 |
| Reflux condenser | |
| Erlenmeyer flask 50 mL | |

### Solutions

| | |
|---|---|
| o-Cresol p.a. | If crystalline, liquefy carefully in a dry box at 35 °C.<br>CAS 95-48-7 |
| Chloroform | CAS 67-66-3 |
| Titrant | c(KOH) = 0.05 mol/L in ethanol<br>CAS 1310-58-3 |
| Bromophenol blue indicator | Dissolve approx. 0.5 g bromophenol blue in 100 mL methanol<br>CAS 115-39-9 |
| o-Cresol/chloroform mixture | In a ratio of 2:3 (v/v) |

### Analysis

*Blank*

The sample preparation is performed as for the PET sample but this time without sample. After the solution has cooled down to room temperature, 0.04 mL bromophenol blue indicator is added and the solution is titrated from yellow to blue with ethanolic KOH.

*Sample*

To the dissolved sample, 0.04 mL bromophenol blue indicator is added and the solution is titrated from yellow to blue with ethanolic KOH.

### Parameters

| | |
|---|---|
| Titration mode | MET U |
| Measurement drift | 50 mV/min |
| Min. waiting time | 0 s |
| Max. waiting time | 26 s |
| Volume increment | 0.01 mL (for blank)<br>0.1 (for titer and sample) |
| Dosing rate | 5 mL/min |
| Pause | 40 s |
| Filling rate | 2.5 mL/min |
| EP criterion | 30 mV |
| EP recognition | greatest |
| Stirring rate | 8 |

### Results

| Carboxyl end groups / (mmol/kg) (n = 5) | s(rel) / % |
|---|---|
| 32.11 | 2.1 |

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