

Application Bulletin



Of interest for:
General analytical laboratories, electroplating, environment

No. 192/1 e

Polarographic and stripping voltammetric determination of thiourea in the lower ppm and ppb range

Summary	Thiourea forms highly insoluble compounds with mercury. The resulting anodic waves are used for the polarographic determination of thiourea. For the analysis of very small quantities (ppb), cathodic adsorption voltammetry (CSV) is used. The DP method is employed in both cases.								
Apparatus	<ul style="list-style-type: none">▶ 2.646.003X VA Processor with 2.647.0020 VA Standor▶ 2.506.0010 Polarecord or 2.626.0010 Polarecord with 2.663.002X VA Stand, eventually 2.608.0010 VA Controller								
Reagents	<ul style="list-style-type: none">▶ Electrolyte solution $c(\text{NaOH}) = 2 \text{ mol/L}$ from NaOH "suprapur"▶ Standard solution $\rho(\text{Thiourea}) = 100 \text{ mg/L}$ as stock solution in dist. water. More dilute solutions must be freshly prepared daily from the stock solution.								
Method	<ul style="list-style-type: none">▶ Range 0.2 ... 2 mg/L thiourea Sample solution (10 mL) and 10 mL electrolyte solution are deaerated well with nitrogen in a polarographic vessel. The DP polarogram is then recorded at the DME between -0.45 V and -0.15 V with a pulse amplitude of $+50 \text{ mV}$. The peak potential of thiourea is at ca. -0.26 V. The content is determined by the standard addition method. After every standard addition, deaerate well with nitrogen.▶ Range 5 ... 60 $\mu\text{g/L}$ thiourea Sample solution (10 mL) and 10 mL electrolyte solution are deaerated well with nitrogen in a polarographic vessel. The stripping voltammogram is then recorded at the HMDE under the following conditions:<table border="0" style="margin-left: 20px;"><tr><td>Electrolysis with stirring</td><td>120 s at -0.2 V</td></tr><tr><td>Electrolysis without stirring</td><td>30 s at -0.2 V</td></tr><tr><td>Voltage sweep</td><td>-0.5 V at -0.9 V</td></tr><tr><td>Pulse amplitude</td><td>-50 mV</td></tr></table> The potential of the stripping peak is at ca. -0.75 V. The content is determined from the (non-linear) calibration curve, which must be set up beforehand.	Electrolysis with stirring	120 s at -0.2 V	Electrolysis without stirring	30 s at -0.2 V	Voltage sweep	-0.5 V at -0.9 V	Pulse amplitude	-50 mV
Electrolysis with stirring	120 s at -0.2 V								
Electrolysis without stirring	30 s at -0.2 V								
Voltage sweep	-0.5 V at -0.9 V								
Pulse amplitude	-50 mV								

Polarographic and stripping voltammetric determination of thiourea in the lower ppm and ppb range

Remarks

- ▶ The largest possible mercury drops should be used ("9" Page 2 646; "3" 663).
- ▶ Chloride ions up to a content of 10 ppm do not interfere with the stripping voltammetry determination.
- ▶ If the sample contains more than 2 mg/L thiourea, it must be diluted for the polarographic determination (non-linear region).
- ▶ More dilute electrolyte solutions or smaller drop sizes restrict the working range. (Neutralise acidic sample solutions beforehand.)

Literature

- ▶ Stara, V. / Kopanica, M.
Adsorptive stripping voltammetric determination of thiourea and its thiourea derivatives.
Anal.Chim.Acta **159**, (1984) 105-110
- ▶ Kirchnerova, J. / Purdy, C.
A new simple voltammetric method for thiourea and thiourea dioxide determinations.
Anal.Lett. **13/12**, (1980) 1031-1040
- ▶ Smyth, M.R. / Osteryoung, J.G.
Determination of some thiourea-containing pesticides by pulse voltammetric methods of analysis.
Anal.Chem. **49**, (1977) 2310-2314

Fig. 1 Program 646 for the polarographic determination

Thioharnstoff ppm-Bereich
MPL 1 EL.TYPE MME

METHOD 18 PAGE 3
OPERATION SEQUENCE

OPERATIONS/PARAMETERS

1	STIR ;PURGE ;	300 s
2	ØPURGE;ØSTIR ;	
3	ØADDL ;	
4	STIR ;PURGE ;	60 s
5	ØSTIR ;ØPURGE;	5 s
6	DME ;MEAS ;	5 s
6a	M.MODE DPN	50 mV
6b	T.STEP	1.0 s
6c	U.SET	-450 mV
7	SWP Ø ;	37 s
7a	U.END	-150 mV
7b	U.STEP	8 mV
	SW.RATE	8.0 mV/s
8	ØMEAS ;ADD132;	5 s
9	BEEP ;END ;	

Polarographic and stripping voltammetric determination of thiourea in the lower ppm and ppb range

Fig. 2 Curve examples 646. Found 0.31 mg/L thiourea by the polarographic method

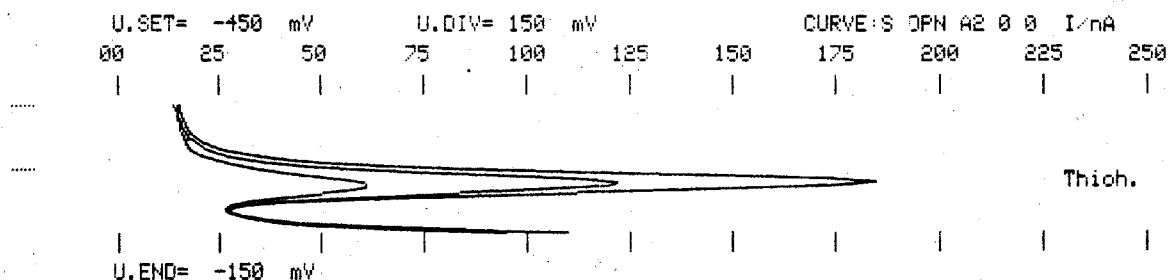


Fig. 3 Program 646 for the stripping voltammetry determination

Thioharnstoff ppb-Bereich	METHOD 30	PAGE 3
MPL 1	EL.TYPE MME	OPERATION SEQUENCE

OPERATIONS/PARAMETERS		OPERATIONS/PARAMETERS	
1	STIR ;PURGE ;	300 s	SW.RATE 8.0 mV/ s
2	BEEP ;HOLD ;		10 MEAS ;REP) 2;
3	(REP. ;		11 BEEP ;END ;
4	PURGE ;STIR ;	30 s	
5	ØPURGE;	10 s	
6	HMDE ;MEAS ;	120 s	
6a	M.MODE	DPN -50 mV	
6b	T.STEP	1.0 s	
6c	U.SET	-200 mV	
7	ØSTIR ;	30 s	
8	MEAS ;		
8a	M.MODE	DPN -50 mV	
8b	T.STEP	1.0 s	
8c	U.SET	-500 mV	
9	SWP Ø ;	50 s	
9a	U.END	-900 mV	
9b	U.STEP	8 mV	

Polarographic and stripping voltammetric determination of thiourea in the lower ppm and ppb range

Fig. 4 Curve example 646.
Found 16.5 $\mu\text{g/L}$ thiourea by the stripping voltammetry method from the calibration curve

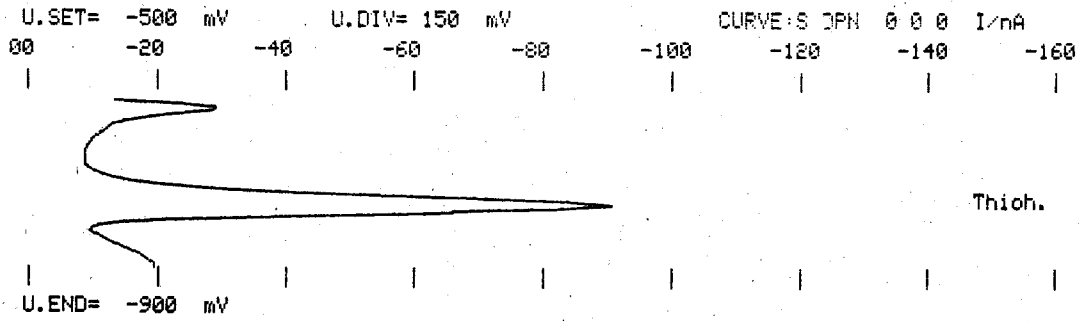


Fig. 5 Calibration curve for the stripping voltammetry method

