MASTER

ASSAY OF TCTNB FOR T_4 CDNB AND TCDNB

C. S. MacDougall

QUALITY DIVISION

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Normal Process Development
Endeavor No. 106

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INTRODUCTION

One of the precursors of TATB is trichlorotrinitrobenzene (TCTNB). Therefore, impurity analysis of the TCTNB for tetrachlorodinitrobenzene (T_4 CDNB) and trichlorodinitrobenzene (TCDNB) is necessary for quality control. A liquid chromatographic technique, developed by W. T. Quinlin of Development Division, was transferred to the Quality Chemistry Lab and adapted to routine production analysis. This note describes the accuracy and precision to be expected for routine assays.

APPARATUS

A Waters ALC-202 is used for the liquid chromatographic analyses. Other instruments of equal competence may be used.

PROCEDURE

CALIBRATION

Standard solutions of T_4 CDNB and TCDNB were prepared by weighing purified materials into spectro grade chloroform. These solutions contained 10 $\mu\text{g}/\mu\text{l}$, 26 $\mu\text{g}/\mu\text{l}$, and 50 $\mu\text{g}/\mu\text{l}$ of each material. Aliquots of each were then injected into a heptane carrier with 0.16% THF. Sample calibration curves are given in Figs. 1 and 2.

SAMPLE ANALYSIS

A 250 mg sample of TCTNB is dissolved in 2 ml of spectro grade chloroform. A 10 μl aliquot is then injected into the chromatograph and peak heights are compared with the calibration curves. Total impurities are then calculated by equation 1.

$$(\%) T_4\text{CDNB} = \frac{\mu\text{g } T_4\text{CDNB (from calibration chart)} \times 100}{1250} \quad (1)$$

The results of the analyses of a test sample having known impurity content are shown in Tables I and II. The T_4 CDNB is essentially 100 percent recovered with a 1.6% relative standard deviation, while TCDNB is 100% recovered with a precision of 10.4% relative standard deviation. Both accuracy and precision are more than adequate.

CONCLUSION

A routine assay capability for TCTNB has been added to the Quality Chemistry Laboratory. Approximately seven samples in duplicate can be analyzed in an eight hour day.

Fig. 1. Total T₄CDNB in TCTNB

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Fig. 2. Total TCDNB in TCTNB

Table I. Precision of Analyses for T₄CDNB and TCDNB

<u>Assay No.</u>	<u>T₄CDNB (%)</u>	<u>TCDNB (%)</u>
1	4.24	1.83
2	4.16	1.92
3	4.16	1.36
4	4.06	1.62
5	4.10	1.81
6	4.18	1.62
7	4.16	1.74
8	4.16	1.74
9	4.02	1.41
10	<u>4.08</u>	<u>1.58</u>
	4.13 ± 0.06	1.66 ± 0.17

Table II. Comparison of Assay Values with Known Values

<u>Component</u>	<u>Quality Assay (%)</u>	<u>Known Value (%)</u>
T ₄ CDNB	4.13	4.02
TCDNB	1.66	1.64

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