2.4.25. ETHYLENE OXIDE AND DIOXAN

The test is intended for the determination of residual ethylene oxide and dioxan in samples soluble in water or dimethylacetamide. For substances that are insoluble or insufficiently soluble in these solvents, the preparation of the sample solution and the head-space conditions to be employed are given in the individual monograph.

Examine by head-space gas chromatography (2.2.28).

A. For samples soluble in or miscible with water, the following procedure may be used.
**2.4.26. N,N-Dimethylaniline**

**METHOD A**

Examine by gas chromatography (2.2.28), using \(N,N\text{-diethylaniline} R\) as the internal standard.

**Internal standard solution.** Dissolve 50 mg of \(N,N\text{-diethylaniline} R\) in 4 ml of 0.1 M hydrochloric acid and dilute to 50 ml with \(water R\). Dilute 1 ml of this solution to 100 ml with \(water R\).

**Test solution.** Dissolve 0.5 g of the substance to be examined in 4 ml of 0.1 M hydrochloric acid. Dilute to 10 ml with water. Dilute 1 ml of this solution to 10 ml with \(water R\). Dilute 1 ml of this solution to 10 ml with \(water R\).

**Verification of precision.** For each pair of injections, calculate for ethylene oxide and for dioxan the difference in area between the peaks of the test solution and reference solution (a). The test is not valid unless the relative standard deviation of the 3 values obtained for ethylene oxide is not greater than 15 per cent and the relative standard deviation of the 3 values obtained for dioxan is not greater than 10 per cent. If the weighings used for the test solution and reference solution differ from 1.00 g by more than 0.5 per cent, the appropriate corrections must be made.

The content of ethylene oxide or dioxan in parts per million is calculated from the expressions:

\[
A_T \times C = \left( A_R \times M_T \right) - \left( A_T \times M_R \right)
\]

\[
D_T \times C = \left( D_R \times M_T \right) - \left( D_T \times M_R \right)
\]

\(A_T\) = area of the peak corresponding to ethylene oxide in the chromatogram obtained with the test solution,

\(A_R\) = area of the peak corresponding to ethylene oxide in the chromatogram obtained with reference solution (a),

\(M_T\) = mass of the substance to be examined in the test solution, in grams,

\(M_R\) = mass of the substance to be examined in the reference solution, in grams,

\(C\) = the amount of ethylene oxide added to reference solution (a), in micrograms.

\(D_T\) = area of the peak corresponding to dioxan in the chromatogram obtained with the test solution,

\(D_R\) = area of the peak corresponding to dioxan in the chromatogram obtained with reference solution (a),

\(C\) = the amount of dioxan added to reference solution (a) in micrograms.