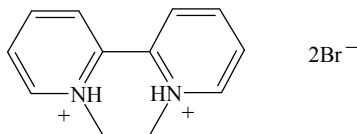


# CIPAC STATUS REPORT

27/06/2005



## 0055 Diquat

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Allocated to GB

CIPAC methods published in :

CIPAC 1, p. 342 (UV)  
CIPAC 1A, p. 1245 (corr.)  
CIPAC E, p. 73 (UV)  
CIPAC E, p. 75 (+ paraquat, UV)  
CIPAC G, p. 47 (Identity tests)

**CIPAC** 15th meeting, October 1971 in Washington

Decision. The methods of analysis (1751) for diquatparaquat water soluble granules, supported by report 1750, are adopted as full CIPAC methods.

**CIPAC** 16th meeting, June 1972 in Stockholm

Decision. The revised method 1874 and the method for the determination of free 2,2'bipyridyl (1874), supported by the report (1873), are adopted as full CIPAC methods.

**CIPAC** 17th meeting, June 1973 in Wageningen

Decision. Diquat dibromide monohydrate is retained as standard, supported by report 1950. Revised method 2070 as full method to be published in 1A.

**CIPAC** 30th meeting, June 1986 in Vienna

The Scandinavian progress report (CIPAC/3316) described the conditions for a HPLC method.

**CIPAC** 38th meeting, July 1994 in Annapolis

Mr Parker introduced an identity test for diquat, CIPAC/3808/m. The test will be published with a reference to the method published in CIPAC E. The specification will have to be republished with a reference to the identity test.

**CIPAC** 51th meeting, June 2007 in Umhlanga Rocks, South Africa

Mr Rodler presented the results on the content of terpyridines on diquat and paraquat formulations.

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## Part 1: Diquat and its SL formulations

FAO specifications are pending. Terpyridines consist of 3 pyridine rings together, with a total of 48 isomers. They are highly toxic (2mg/kg oral), and they chelate with transition metals to form stable complexes.

During the manufacture of diquat, terpyridines are produced as impurities. Here, a method development is described for the determination of terpyridines in diquat and paraquat TKs and their SL formulations. Extraction is followed by two SPE clean-up steps, with subsequent analysis by LC-MS/MS.

### Peer review summary

Two Syngenta laboratories and the CSL Laboratory in UK participated, testing Syngenta diquat TK, Syngenta diquat SL formulations, Syngenta diquat/paraquat mixed SL formulation, and non-Syngenta diquat SL formulation. Terpyridines were found to be unstable in sample solutions.

Recoveries of terpyridines in the diquat TK were acceptable for the one Syngenta laboratory and the CSL Laboratory but low for the US Syngenta Laboratory. The recoveries for diquat/paraquat mixed formulation were acceptable.

The method works well for the diquat TK, diquat SL formulations and diquat/paraquat SL formulations. The SPE clean-up is very reliable and very effective, as co-extractive material is removed so that the need for matrix-matched calibration solutions is not required.

LC-MS/MS under the conditions used shows high sensitivity to terpyridines, and enables the determination of terpyridine at low levels.

It is recommended to adopt this method for determination of terpyridines in diquat TK and diquat SL formulations as part of the FAO specification.

### Decision

The independent laboratory validation of the HPLC-tandem mass spectrometry method for determination of residues of the relevant impurity terpyridines in diquat TK and SL was noticed and regarded to be suitable for the determination of the relevant impurity terpyridines in TK and SL, subject to clarification of certain points.