

## NATIONAL RESEARCH CENTRE FOR GRAPES, PUNE

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### **National Referral Laboratory for monitoring pesticide residues for export of table grapes from India to EU countries (2009-10)**

This was the Seventh year of the Residue Monitoring Plan, initiated by the APEDA, Ministry of Commerce, Government of India in 2003-04 with National Research Centre for Grapes, Pune through the National Referral Laboratory (NRL) setup under this institute. The NRL was initially set up for monitoring of pesticide residues in table grapes. From this year, its scope was expanded to cover all important fruits and vegetables.

Presently three state Governments viz. Maharashtra, Andhra Pradesh and Karnataka are covered under this plan since exportable grapes are produced in these states. The farms from where table grapes are to be exported are registered with the Agricultural Departments of the respective grape growing states. The total registered farms for export of table grapes as per our records in these states were 14930. Out of these farms, 14704 farms were from Maharashtra alone. The total area registered for export of table grapes was about 10189.585 hectares as against the estimated acreage of 64,300 hectare in the country.

Under the Pesticide Residue Monitoring Plan, ten APEDA-nominated laboratories viz. Vimta Labs Ltd. (Hyderabad), Reliable Laboratories (Thane), Geo-Chem Laboratories (Mumbai), Doctors Analytical Laboratories Pvt. Ltd. (Pune), Shriram Institute for Industrial Research (Bangalore), SGS laboratories (Chennai), National Horticulture Research and Development Foundation (NHRDF, Nashik), Delhi Test House (Delhi), Interfield Laboratories (Kochi), Arbro Pharmaceuticals Ltd. (Delhi) actively participated in drawing of samples and residue monitoring. In addition, four more nominated laboratories viz. Pesticide Residue Testing Laboratory (Pune), True Analytica (Chennai), Sargam Laboratory (Chennai), Shiva Analyticals (Bangalore) participated in the proficiency test (PT) rounds organized by the NRL, but they did not analyze any export grape samples under the Grapenet in this year.

### **Proficiency test**

The first Proficiency Testing (PT) round among the nominated laboratories was organized on three commodities viz. grape, tomato and pomegranate. In the first PT round the performance of only six laboratories was satisfactory in terms of identification of spiked pesticides and the corresponding 'Z'-scores and they were immediately allowed by the NRL for access into the Grapenet software system. A second PT round was organized and in this PT, the performance of all the laboratories were overall satisfactory, with few exceptions where specific laboratories failed to result in satisfactory 'Z' score within the prescribed limit of -2 to +2. The failure of a few laboratories in this test also was found to be inaccurate calibration standards and corresponding erroneous calibration graphs used by the laboratories for calculation of the PT results. These laboratories were asked to re-validate the entire methodology with fresh matrix-matched. The nominated laboratories were only allowed to participate in residue monitoring in registered field samples once the NRL got satisfied with their validation results.

## **FAPAS proficiency testing program**

During this year, the NRL initiated the participation of the nominated laboratories in International proficiency testing program on grape puree organized by the FAPAS (Food Analysis Performance Assessment Scheme), Government of UK. All the 14 nominated laboratories and the NRL participated in this PT round. All the nominated laboratories could successfully identify each target analyte. The results in terms of “Z-score” were satisfactory for all the target pesticides except for few deviations for propargite, which could have happened due to its degradation during long transit time between UK and India.

## **Research activities:**

In last one year, the NRL could expand the scope of the multiresidue analysis method to cover testing of more than 200 pesticides in various fruits and vegetable matrices viz. grape, pomegranate, mango, apple, orange, onion, etc. on GC-MS and LC-MS/MS. A simple single residue method has been developed and validated for the analysis of the fungicide dinocap (and meptyldinocap) and the antibiotic viz. aureofungin. The tandem mass spectrometric method on GC-MS/MS for high sensitivity analysis of synthetic pyrethroid group of pesticides was standardized. Besides, the NRL developed an effective technique of residue analysis of problematic pesticides like captan, captafol, iprodione, etc, which avoided expensive cryogenic crushing as practiced in Europe and the results were comparable to cryogenic crushing. The NRL also standardized a sensitive LC-MS/MS based multiresidue method for estimation of plant growth regulators including gibberellic acid, NAA, Chlormequat chloride, etc.

## **Monitoring results:**

A total of 5009 table grape samples were tested in the 2009-10 season by ten nominated laboratories, which include first sample as well as resample. Out of the 5009 total analyzed samples, 501 samples failed for EU-MRL compliance. Thus, a total 501 internal alerts were issued. On re-sampling after the recommended pre-harvest intervals, 187 alerts were subsequently revoked on the basis of the MRL compliance in analyses reports.

In totality, there were 34 pesticides for which MRL exceedances were recorded in the 2010 season. Most frequently detected insecticide was Captan with 150 detections. The other major insecticides that got detected in this season include abamectin with 76 detections and the other pesticides viz. lambda-Cyhalothrin and chlorpyrifos which were detected in 50 and 47 samples, respectively. In case of the fungicides, highest detected chemical was flusilazole with 59 detections followed by carbendazim with 31 detections.

Out of the 314 effective alerts, which accounts for 7.46% of the samples analyzed, most of the cases correspond to those pesticides, which are mostly used during the last two months before harvest. Hence, the management of these pests before harvest will certainly play a key role in minimizing the residues of pesticides in next grape season of 2010-11. The detections of the non-recommended chemicals indicate increasing awareness among the grape growers to use the non-recommended chemicals for pest management.

### **Detection of Chlormequat chloride residues in Indian grapes for export to EU:**

The residues of the plant growth retardant viz. Chlormequat chloride (CCC) was reported in Indian grapes from Europe in this season, which emerged as a major issue at the end of the current grape season. On receipt of the information regarding detection of CCC in Indian grapes at above the EU MRL of 0.05 mg/kg in a laboratory of Sweden on 12<sup>th</sup> April, extensive monitoring of CCC residues in India was initiated under the leadership of the NRL since 15<sup>th</sup> April. All the grape consignments yet to be exported were screened compulsorily for CCC residues.

A total 454 export grape samples were thus tested for the residues of Chlormequat chloride during April-May 2010 by the nominated laboratories and the NRL. Out of the tested samples, 306 samples conformed to the EU-MRL of 0.05 mg/kg, while 148 samples failed for EU-MRL compliance which accounts for a failure percentage of 32.6%. On domestic front, in comparison to the MRL specified under the PFA Act of the Ministry of Health and Family Welfare, Govt. of India, however, 450 out of the total 454 tested samples complied with the Indian food safety regulations.

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