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## The Quality Control Facilities and the Upgraded Know-how Provided by the Project

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**SUMMARY** – The specialists' expert support to the GCSAR lab facilities specialized both in olive oil and table olive analyses is described. Dedicated activities have been carried out in order to upgrade technology, infrastructures, hardware and software with a network connection for data transfer and elaboration. Training activities have been performed all along the project duration (2 and half years) in Italy at Chemiservice, where 4 Engineers of GCSAR have been trained for olive oil analyses during 162 man days and at CRB, where 2 engineers of GCSAR Olive Department (Idleb) learned during 234 man days the main operations concerning the production and quality control of table olives. They have also acquired information about alkaline methods and the natural fermentation for both black and green olives.

Two technical laboratories for oil analyses and for following up processing and analyses of table olive (chemical and microbiology analyses) have been established, upgraded and updated at GCSAR Olive Department of Idleb.

One panel test room with annexed preparation room (8 seats) for virgin olive oil tasting is now operational in full conformity with EU standards.

**Key words:** Implementation, laboratory, olive oil, table olives, chemical and panel tests, training

**RESUME** - Les experts italiens spécialistes ont supporté avec leur expérience les structures du CGRSA qui depuis long temps avaient été dédiés aux analyses des olives de table et des huiles d'olive. Les activités ont été réalisées pour mettre à jour, moderniser les structures pré - existants et former aussi le personnel technique des labos.

Les 4 Ingénieurs du CGRSA responsables des analyses des huiles d'olive ont été formés auprès du Chemiservice pendant 162 jours/homme, lorsque 2 Ingénieurs spécialistes en analyses et préparation des olives de table ont été formés pendant une période complexe de 234 jours/homme au CRB, ou les ingénieurs du CGRSA ont appris les opérations fondamentales sur le control de qualité des olives de table et les analyses principales à effectuer sur celles-ci.

Ils ont aussi appris les méthodes pour le traitement avec sode caustique et la fermentation naturelle pour les olive "en vertes " et " en noir".

Deux labos ont été mis en place auprès de la structure du CGRSA d'Idleb. Les labo ont été restaurés, mis en sécurité et reformés. Un labo pour les analyses chimiques sur les huiles vierges d'olives a été réalisé et un'autre labo sur l'analyses du processus de fermentation et pasteurisation des olives de table avec aussi les instruments et les accessoires pour executer les analyses chimiques et biologiques nécessaires pour le control de qualité des olives de table.

**Mots clés:** Réalisation, mis à jour, laboratoire huile olive, laboratoire olives de table, panel test, test de laboratoires, formation

### Introduction

Originally the GCSAR laboratory in Idleb was organized by Syrian government in the framework of a programme aiming at providing the national companies involved in table olives and olive oil production and trading with a quality support service.

The table olive laboratory was in the basement and just pH and salinity concentration analyses were carried out.

The oil laboratory had two main tasks. On the one hand, they studied Syrian olive varieties to assess the conversion of olives in virgin olive oils (processing technology, oil output rating in national cultivars, definition of standards for the assessment of the right ripening degree before picking); on the other hand, the laboratory worked for a better definition chemical-physical and organoleptic characteristics of the oils produced in the national territory. This task aimed at providing olive oil workers (producers and packaging operators) with product classification data according to the criteria fixed by international standards in order to facilitate marketing and export.

However, GCSAR laboratories potential was not commensurate enough with their tasks because of structural and instrumental lack; replies to customers were not given in time and didn't comply with all the necessary formalities.

The project for the "Technical assistance for the improvement of the olive oil quality in Syria" production, funded by Italian government, has carried out a full upgrade and update programme aiming to restructure the pre-existing laboratories both for olive oil and table olives quality analyses, increasing knowledge, equipment, technology and instruments. The main purpose was to achieve the organizational and qualitative levels needed to finally acquire ISO 17025 laboratory certification.

### **Project related activities**

In order to achieve the aforesaid goals, the pre-existing laboratories have been visited by Italian experts aiming at defining room reorganization and assessing operational skills.

On the basis of checking results, the following interventions were decided:

- to arrange the rooms on 2 floors from a structural point of view in order to make them fit for use and safe as chemical and microbiological laboratory;
- to send some laboratory technical personnel to Italy for a two month formative stage at a chemical laboratory specialized in olive oil analytical tests, certified and recognized by international authorities;
- to buy new laboratory instruments and accessories to complete the equipment and expand the potential;
- to provide the laboratory with suitable hardware and software for efficient data management and the achievement of prompt certification and data recording;
- to carry out all interventions needed to include the laboratory in international chains in order to obtain the recognition by protection authorities;
- to create the conditions for the laboratory ISO 17025 certification.

During laboratory room restoration and adjustment, five technicians already working in the aforementioned building were sent to Italy for full immersion stage at specialized laboratory Chemiservice S.a.s based in Monopoli.

At this laboratory, it was possible to deepen new laboratory techniques to carry out all analyses required by International Olive Oil Council (IOOC) Trade Standard and by EC law for the determination of olive oil quality and table olives genuineness.

Syrian technicians studied particularly how to perform the following analyses on olive oil :

- free acidity
- peroxide value
- absorbency in ultra-violet
- fatty acid composition

- sterol composition
- erythrodiol+uvaol
- waxes
- stigmastadiens
- saturated fatty acid content in the 2-position
- aliphatic alcohols
- solvent residues
- ecn-42 triglycerides
- polyphenols

During the stage at Chemiservice Syrian technicians were intensively trained for carrying out the aforesaid tests. They also investigated all theoretical and practical issues concerning those tests together with technicians working at Chemiservice.

Tests of specific oil samples coming from different Syrian production areas and cultivars were performed in order to define better the issue related to a specific anomaly of Syrian olive oils that show very often the delta 7 stigmasterol value higher than the allowed threshold (max 0.5%).

The results of those experiments came under further studies and closer examination and were discussed during different congresses and chemists' meetings of International Olive Council.

After that experience, in their new Syrian laboratory that at that time was complete and ready to offer new practical chances, the three technicians could apply what they learned in Italy.

Actually during the use of new equipment and the performance of new tests, expected issues appeared at the laboratory in Idleb.

In the beginning, these issues were discussed by usual information systems, later they asked for an Italian technician working at Chemiservice laboratory to help more effectively.

During this calendar year two operative missions followed one another: two Syrian technicians came back to Chemiservice laboratory in Italy and two experts from Chemiservice went to Syria for the final inspection of the laboratory in Idleb.



Photo 1 and 2: Research activities in the new olive oil laboratory

We should dwell upon the last mission to describe accurately its course and mainly to consider it as the starting point of future ameliorative interventions.

The aim of that mission was to verify the level of efficiency of the new laboratory made to carry out physical-chemical analyses on sample of olive oils produced in Syria.

We coped with the issues that had emerged during the first period of the running of the laboratory,

we verified in particular:

- i) state of the actual equipment functionality (3 GC, 1 HPLC, 1 GC/MS);
- ii) type of gas chromatography columns used;
- iii) quality of chromatograms and data sheets;
- iv) availability of reagents and standards needed to perform the analyses;
- v) availability of glassware and consumables;
- vi) functionality of the computer program for data management.

The delivery and the subsequent analyses of some oil samples already analysed by Italian laboratory Chemiservice were also planned.

After the first visit of the laboratory and the examination of the general state of equipment and accessories, the HPLC was examined checking the operative conditions during use (flow, column temperature, type of column and eluant, state of I.R. detector). A set of tests was also performed to identify the operative parameters needed to obtain more efficient in triglyceride analysis.

Computing system was verified too and a new computer program for the management of the new GC and GC/MS instruments was reinstalled.

Vacuum condition was restored in the quadrupole of the GC/MS instrument. Once that equipment was reset, all instruments useful for oil analyses were running regularly, baselines were adjusted and interferences were removed. We intervened in liner cleaning and verified their response after standard insertion.

We also verified the procedures for the checking of the preparation step for olive oil analyses.

The following interventions were carried out:

- checking of the preparation step of the method for the assessment of methyl esters of fatty acids;
- checking of the preparation step of the method for the assessment of sterol composition;
- checking of the preparation step of the method for the assessment of total waxes;
- checking of the preparation step of the method for the assessment of ECN 42 difference;
- checking of the preparation step of the method for the assessment of stigmastadiens.

To complete the plans, laboratory tests were performed on three oil samples already analysed by Chemiservice in order to compare the results and verify that they were in accordance with IOOC Trade Standard.

Those checks were carried out together with Syrian technical staff working at the laboratory.

In the end, changes were made (through the agency of the selling company) in some files of EUROSOFT computer program, purchased for the laboratory data management. The gas chromatographic column was installed on GC/MS system, the air pump was put in working order, the software was installed, the parameters needed to perform the analyses in SIM and SCAN were defined, the instrument auto-tune was checked using a polycyclic aromatic hydrocarbon standard solution.

Concerning the table olive laboratory, a dedicated training activity has been performed in Italy at Centro Ricerche Bonomo – Castel del Monte – Andria (BA), where two engineers learned the main operations concerning the process and quality control of the table olive. First of all, they acquired information about alkaline methods for the reduction of bitter taste and then they applied many times the same method to cv “Bella di Cerignola” (green table olive produced in South Italy) (Brighigna, 1998) with different sizes. Moreover they applied dehydration in forced air to black olive cv “Pasola” and studied the natural fermentation for both black cv “Caroleo” and green olives cv “Bella di Cerignola”. Finally they learned how to pack and pasteurize the final products in sealed glass jars. In particular we can cite the main issues as follows:

- i) the trade classification of fresh table olives, according to the International Olive Oil Council (IOOC, 2004), defines three types of olive: Green olives (harvested to maximum size and before they change green colour), Olives turning colour (harvested before the full ripeness), Black olives (harvested fully ripe);
- ii) the trade classification of processed table olives, according to the Qualitative Standard of Table Olives in IOOC, defines the following ways of marketing: Whole (the fruits keep their original shape), Stoned (the olives maintain their original shape, without stone), Stuffed (stoned olives filled with different vegetables), Halves (stoned or filled olives cut in two halves), Quarters (stoned olives cut in quarters), Segments (stoned olives cut in more than four pieces), Slices (stoned or stuffed olives cut in slices), Pieces (not definite small pieces of stoned olives), Olive paste (table olives fermented and ground), Olives for salad (broken stoned fruit), Olives with capers (whole or stoned fruit, generally of small size, with capers and peppers) ;
- iii) the quality of processed table olives is related to the following parameters of fresh product: colour, fruit dimension, pulp/stone weight ratio (more than 3/1), sugar content (more than 4%), texture (crispy with thin skin), appearance (no damage, browning, spoilage), easy separation of the stone from the pulp. These characteristics are related to the cultivar and they change during the process. This implies that process parameters (lye concentration, timing, temperatures, percentage salt in fermentation and in cover solution, etc.) are different for each cultivar;
- iv) the processed table olives must be uniform in colour and shape, unblemished, clean, without off flavours and off odours caused by anomalous fermentation. Furthermore, they must be free from any harmful bacteria and toxins. The olives can be classified into: Extra (best quality), First class (good quality), Second class (other fruits);
- v) the best debittering method (Sevillian) has been applied in the pilot plant of CRB on green Bella of Cerignola. The olives were pre-treated with a lye solution to remove the bitter taste (oleuropein) and preserved in brine until the completion of natural lactic fermentation. During the process the engineers learned to characterize the olive samples with the following analyses: lye penetration, olive pH, salt and sugar content, lactobacillus and total bacteria count;
- vi) the cv Pasola has been dehydrated in forced air after blanching pre-treatment in lye solution (0,3% NaOH) at 90°C and after salting (10% NaCl). The drying process has been carried out in a dryer cabinet of CRB with air at 50°C. The final product has been packed in Modified Atmosphere (10% O<sub>2</sub> and 90% N<sub>2</sub>).



Photo 3: Research activity in the table olive laboratory

### Set up of a table olive laboratory

A technical section for the processing of table olives was created in GCSAR Olive Department - Idleb. The laboratory was supplied with the following equipment: processing tanks, digital thermometers with data logger, electric pasteurizer for manual and automatic operations, refrigerators, freezer, growing rooms for microbiology tests, hood with laminar flux, electronic colony count and other generic instruments.

## Conclusions

At the end of the project briefly described above, a positive operational result was achieved thanks to all participants.

We hold the view that the laboratory in Idleb is currently enabled to perform the analyses requested by IOOC Trade Standard complying with all the necessary formalities and beating the clock according to customers' needs. The Syrian laboratory technicians have been well trained to acquire more reliability and prestige. To keep and implement the gained experience, we suggest:

- to guide the laboratory to ISO 17025 certification having recourse to local professional advice or accredited laboratories;
- to accredit all chemical tests and sensory analysis required by international standards for olive oil product classification;
- to deepen the use of analytical techniques in GC/MS and LC/MS for pesticide and contaminant analyses through personnel attendance at educational classes in Syria or abroad;
- to participate in ring tests with other laboratories at least annually to verify data reproducibility;
- to maintain contact with other laboratories and get external professional advice in order to acquire new knowledge and the needed updates;
- to meet table olive producers in order to share information about any problems;
- to implement a new marketing section in order to direct the technicians according to the consumer expectations.