

Tuberose Concrete Fractionation by Supercritical Carbon Dioxide

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Supercritical CO₂ extraction was used to postprocess a solid tuberose extract that was produced by liquid solvent extraction. The optimum extraction conditions to produce the tuberose volatile oil were found operating at 80 bar and 40 °C. At these conditions no undesired compounds have been coextracted except waxes. These latter compounds have been eliminated by adopting a stagewise separation procedure consisting of two separators operated in series. The first separator was set at 80 bar and -5 °C to allow the precipitation of waxes. The second separator operated at 15 bar and 0 °C to recover tuberose volatile compounds. The oil yield was 24% by weight of the charged material. The process evolution has been monitored by performing GC-MS analysis of the extracts. A large modification in the composition of tuberose volatile oil has been observed with the increase of the extraction time.

Keywords: *Supercritical extraction; tuberose; concrete; fractionation*

INTRODUCTION

Essential oils obtained from flowers have, as a rule, a high commercial value. In several cases, flowers have to be treated in an early stage to avoid the degradation of their fragrance. The conventional methods used to obtain flower extracts are steam distillation, solvent extraction, and "enfleurage". In the case of tuberose, the yield of the steam distillation process is very low and does not reproduce the characteristic fragrance of the flowers (Fenaroli, 1963). Therefore, the extraction with a volatile solvent or with the enfleurage technique is preferred. In the case of solvent extraction, the subsequent solvent vaporization produces a quasi-solid product called "concrete" which contains the fragrance compounds but also a high percentage of undesired compounds, mainly paraffins belonging to cuticular waxes covering the surface of flowers.

Since many industrial applications require liquid products containing only the fragrance compounds, a concrete has to be further processed. A typical treatment is the solubilization of concrete in ethyl alcohol and the elimination of waxes by cooling the solution (Anac, 1986). The product obtained by this technique is called "absolute". The absolute contains all the fragrance compounds, but sometimes also fatty acid methyl esters and some paraffins. As reported by La Face (1952) in the case of tuberose, the volatile oil obtained by absolute distillation yielded only 6.2% by weight of the absolute processed. Tuberose concrete can also be directly treated by steam distillation to obtain a "volatile oil" with a yield from 3 to 6% by weight of the starting material. Steam distilled oil from concrete contains a very low percentage of methyl antranilate (La Face, 1952).

The supercritical CO₂ extraction performed directly on tuberose flower is not applicable on the industrial

scale since the yield in essential oil is less than 0.1% by weight. For example, some authors (Moates and Reynolds, 1991; Vidal and Richards, 1987) attempted the liquid and supercritical CO₂ extraction of essential oil directly from rose petals. Also in the case of rose flowers the content in essential oil is less than 0.1% by weight and these authors obtained a semisolid extract with low yields (0.07–0.08% by weight) and a high content of coextracted paraffins. Therefore, using this process, it should be necessary to use a very large supercritical fluid extraction plant to obtain an acceptable industrial production of volatile oil.

The postprocessing of a flower concrete was first attempted by Gopalakrishnan and Narayanan (1991), who fractionated the jasmine concrete using liquid CO₂ operating at 100 bar and at 20 °C. The use of liquid CO₂ and of a single-stage separation gave an incomplete elimination of paraffins from the volatile oil.

In this work, we tried a tuberose concrete fractionation by supercritical CO₂ with the aim of separating volatile oil from the higher molecular weight compounds. The extraction process was coupled to a fractional separation technique that uses two separation stages operating in series (Reverchon et al., 1995a,b). GC-MS analyses of the extracts were proposed, and the process optimization was attempted with respect to the extract composition and yield.

MATERIALS AND METHODS

Apparatus and Procedures. The supercritical fluid extraction (SFE) apparatus used in this work was mainly formed by an extraction vessel with an internal volume of 200 cm³. A high-pressure pump (Milton Roy, mod. Milroyal B, Point Saint Pierre, France) capable of a maximum pressure of 500 bar was used to deliver the solvent. The two separators had an internal volume of 200 cm³ and can be used at a maximum pressure of 300 bar. The apparatus has been described in detail elsewhere (Reverchon et al., 1995a).

In each test, about 27 g of tuberose concrete was warmed to 30 °C, mixed with 270 g of glass beads (2 mm diameter), and then charged in the extractor (ratio concrete/glass beads

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≈ 0.1). This procedure was used to maximize the contact surface between the solute and the supercritical solvent and to avoid the formation of preferential solvent channels into in the extractor. The solution at the exit of the extractor went through the two separators in series to separate volatile oil from waxes. A pressure of 80 bar and a temperature of -5 °C were set in the first separator, and a pressure of 15 bar and a temperature of 0 °C were fixed in the second one. These conditions were chosen on the basis of previous studies on fractional separation processes (Reverchon et al., 1995a) to obtain an efficient precipitation of the waxes in the first separator and to minimize the loss of volatile compounds in the gaseous CO₂ stream at the exit of the apparatus.

The asymptotic yield in volatile oil was of 24% by weight. Different CO₂ flow rates were also used during the experiments (0.8, 1.2, and 1.6 kg/h, respectively). They do not affected the extraction yield but only the extraction rate. They were also used in the mathematical modeling of the process (Reverchon and Poletto, 1996).

Materials. Indian tuberoso concrete (*Nepeta tuberosa* L.) was supplied by Sanofi (Grasse, France). Concrete was semisolid with a dark orange color. It was spreaded over a glass slide and observed by an optical microscope with a contrast phase condenser. It appeared to be constituted by a crystalline phase (waxes) and an amorphous phase (oil) adsorbed on it.

CO₂ (99.9% purity) was supplied by SON (Società Ossigeno Napoli, Italy).

Ethyl Alcohol Extraction of Concrete. Tuberoso concrete (1 g) was dissolved in 19 g of anhydrous ethyl alcohol at 30 °C. The solution obtained was cooled at -10 °C for 1 min and then spin-dried for 1 h at 800 rpm. The upper part of the solution, clear and with a light yellow color, was recovered after ultrafiltration and cooled for 1 h to precipitate the remaining waxes and then spin-dried for another 1 h. This last procedure was repeated five times to ensure an efficient precipitation of waxes. The clear ethanol extract was analyzed by GC-MS. The yield of tuberoso absolute was 11% by weight of the starting material. This procedure is similar to the one used in the industrial preparation of commercial absolutes (Anac, 1986).

Gas Chromatography-Mass Spectrometry (GC-MS). The GC-MS apparatus was a Varian (San Fernando, CA) model 3400 gas chromatograph equipped with a fused-silica column DB-5 (J&W Scientific, Folsom, CA), 30 m × 0.25 mm i.d., film thickness 0.25 μm. The GC apparatus was interfaced with an ion trap mass spectrometric detector (ITS 40, "Magnum", Finnigan Mat, San Jose, CA). The GC conditions were as follows: oven temperature of 50 °C for 5 min, then a programmed increase from 50 to 250 °C at 2 °C/min, and a fixed isothermal hold at 250 °C for 60 min.

The sum of the area percentage of compound families in which tuberoso volatile oil was divided (see Figure 2) was calculated from the gas chromatographic traces and then converted into an absolute value using ion trap relative response factors. These corrected area percentages were also used to calculate the yield of the compound families reported in Figure 3. The response factors were estimated using standard compounds having the same molecular weight of the compound families that constituted the tuberoso oil. In particular, we used limonene, cineole, eugenol, caryophyllene, *n*-heicosanol, and linear paraffins from C20 to C30.

The compounds extracted were identified by comparing their retention times and mass spectra with those of pure reference compounds. Mass spectra were also compared with those in the NIST (National Institute of Standards and Technology) and WILEY5 mass spectra libraries.

RESULTS AND DISCUSSION

GC-MS analysis has been used to identify the compounds forming tuberoso concrete. The chemical characterization of concrete in terms of compounds families also provided indications on the conditions to be adopted during the supercritical fractionation.

Table 1. Comparison among the Composition of the Starting Material (Concrete Column) and of the Various Fractions Collected in the Second Separator Operating at 80 bar, 40 °C^a

compound ^b	concrete (%) ^c	F 1 (%) ^c	F 2 (%) ^c	F 3 (%) ^c	HP (%) ^c	waxes (%) ^c
benzaldehyde	0.03	0.42	0.37	0.60	0.45	—
β-pinene	0.26	1.15	0.08	0.18	0.47	—
<i>p</i> -cymene	0.13	0.19	0.04	—	0.76	—
1,8-cineole	3.41	8.22	0.25	0.09	0.18	—
<i>p</i> -cymenene	tr ^d	0.25	0.05	—	—	—
methyl benzoate	3.57	16.47	0.26	0.12	—	—
2-methylbenzoxirone	0.24	1.13	0.11	—	—	—
benzyl acetate	tr	0.35	0.05	—	—	—
α-terpineol	tr	1.25	0.78	0.15	0.17	—
methyl salicylate	3.57	6.68	0.22	0.03	—	—
ethyl benzoate	0.17	0.49	0.20	0.24	0.15	—
phenyl butylacetate	0.22	0.08	0.05	—	—	—
methylanthranilate	0.66	0.52	1.36	0.76	0.15	—
benzyl butyrate	0.07	0.24	0.07	—	—	—
eugenol	0.48	1.13	0.97	0.14	—	—
β-bourbonene	tr	0.31	0.09	tr	—	—
vanillin	tr	tr	tr	0.24	—	—
methyleugenol	1.07	2.61	2.00	0.50	0.22	—
caryophyllene	0.11	0.31	0.15	0.07	—	—
β-gurjunene	0.04	0.17	0.10	0.05	—	—
<i>cis</i> -isoeugenol	tr	3.33	7.52	2.03	—	—
<i>cis</i> -methylisoeugenol	2.73	0.46	0.54	0.61	0.77	—
<i>cis</i> -β-farnesene	0.30	tr	tr	tr	1.05	—
valencene	2.87	4.93	4.92	1.63	0.72	—
nepetalactone	4.08	4.34	10.10	14.18	13.47	—
dehydronepetalactone	0.90	0.80	2.98	3.23	1.93	—
<i>trans</i> -methylisoeugenol	15.21	31.89	44.79	23.88	9.44	—
α-farnesene	1.44	3.08	2.77	0.85	0.58	—
α-farnesol	0.90	—	—	2.33	6.54	—
eugenyl acetate	6.09	8.15	15.75	36.701	18.64	—
benzyl benzoate	0.72	0.40	1.50	4.06	2.64	—
C ₁₄ H ₁₂ O ₂	0.81	—	1.93	5.90	1.96	—
<i>n</i> -octadecan-1-ol	tr	—	—	—	0.46	—
<i>n</i> -nonadecane	0.02	0.11	—	0.09	3.75	tr
compd not id. (MW 220)	0.06	—	—	0.35	0.30	—
<i>n</i> -heicosane	0.01	—	—	—	0.39	tr
<i>n</i> -heneicosane	0.08	0.18	—	0.14	4.91	tr
<i>n</i> -tricosane	0.90	0.31	—	0.34	4.88	2.29
<i>n</i> -tetracosane	0.39	—	—	—	—	0.94
<i>n</i> -tricosan-1-ol	0.36	—	—	—	2.40	—
<i>n</i> -pentacosane	10.79	0.96	—	0.52	0.91	30.69
<i>n</i> -hexacosane	0.98	—	—	—	—	3.02
<i>n</i> -tetracosan-1-ol	1.52	—	—	—	13.32	—
<i>n</i> -heptacosane	20.25	—	—	—	—	43.37
<i>n</i> -octacosane	0.61	—	—	—	—	1.20
<i>n</i> -pentacosan-1-ol	1.62	—	—	—	8.38	0.31
<i>n</i> -nonacosane	10.91	—	—	—	—	14.97
<i>n</i> -hentriacontane	1.46	—	—	—	—	3.20

^a The fractions were recovered after 20 min of extraction (F 1 column), in the extraction time interval between 360 and 480 min (F 2 column), and in the extraction time interval between 690 and 750 min (F 3 column), respectively. The composition of the fraction collected in the second separator after an extraction performed at 100 bar and 40 °C (HP column) and of the product recovered in the first separator at the end of an extraction test (Waxes column) were also reported. ^b The compounds are listed in elution order on a DB-5 column. ^c Area percentages were reported without correction factor. ^d tr < 0.01%.

The detailed identification of compounds forming the tuberoso concrete is reported in Table 1 (concrete column). It was constituted by an area percentage of 50.1% of fragrance compounds, with *trans*-methylisoeugenol (15.2%) and eugenyl acetate (6.1%) as the major compounds. Two lactones are also responsible for the tuberoso aroma: nepetalactone and dehydronepetalactone (Cotrim et al., 1994). They have been found in an overall percentage of 5.0% in the tuberoso concrete. Waxes (coextracted with the aroma compounds by hexane) constituted 46.4% of the concrete, giving it a semisolid aspect. A percentage of 3.5% of long-chain alcohols was also present: they can adversely affect tuberoso fragrance (Fenaroli, 1964).

This information on chemical composition of the tuberoso concrete confirmed that it is necessary to optimize the selectivity of supercritical CO₂ to obtain a proper tuberoso concrete fractionation. Indeed, it is necessary to extract only the compounds responsible for the fragrance by leaving the other compounds unextracted. Furthermore, a fractional separation technique has been used to selectively precipitate the paraffins that were solubilized by supercritical CO₂ during the extraction process (Reverchon et al., 1995a,b). Therefore, systematic SFE tests have been performed in the range from 80 to 100 bar operating at a temperature of 40 °C. We analyzed the product collected in the two separators by GC-MS. By operating at 80 bar, we obtained the maximum content of fragrance compounds in the extract collected in the second separator.

Since the different compound families that constitute the tuberoso volatile oil (hydrocarbon terpenes, oxygenated terpenes, and benzene derivatives) showed different extraction rates, the compositions of the tuberoso oil changed during the extraction process. To monitor the variation of the extract composition during the extraction process, we collected the oil at fixed time intervals (every 20 min) and analyzed the various fractions by GC-MS. In Figure 1, we reported three GC traces of tuberoso volatile oil recovered in the second separator after 20 min of extraction (A), from 360 to 480 min of extraction (B) and from 690 to 750 min of extraction (C). From this figure, it is possible to perform a qualitative evaluation of the progressive shift of the extract toward compounds with higher molecular weight. Results reported in Table 1 give a quantitative representation of the composition changes during extraction. Indeed, the extract recovered after 20 min (F1 column) contains a percentage of 83.8% of oxygenated compounds (monoterpenes and benzene derivatives), with the main contributions coming from 1,8-cineole (8.2%), methyl benzoate (16.5%), methyl salicylate (6.7%), *trans*-methylisoeugenol (31.9%), and eugenyl acetate (8.1%). Lactones (5.1%) and sesquiterpenes (8.8%) were also found. The volatile fraction recovered in extraction time interval between 360 and 480 min (F2 column, Table 1) consists of 78.7% of oxygenated compounds with an increment of the percentage of the *trans*-methylisoeugenol (44.8%) and of the eugenyl acetate (15.8%), whereas the most volatile compounds such as methyl benzoate and methyl salicylate were reduced to a percentage of 0.3 and 0.2%, respectively. An increment of the lactones percentage (13.1%) was also observed. The fraction recovered in extraction time interval between 690 and 750 min (F3 column, Table 1) contained very low quantities of aroma compounds (only the 0.1% of methyl benzoate and traces of methyl salicylate), but still large quantities of *trans*-methylisoeugenol (23.9%), eugenyl acetate (36.7%) and lactones (15.8%). Some paraffins were also present though in a low percentage (1.1%). The composition change of the volatile oil during the whole extraction process is shown in Figure 2, where the area percentage of the different compound families against the extraction time is reported. Tuberoso oil compounds have been divided in three families: hydrocarbon compounds (monoterpenes and sesquiterpenes), oxygenated compounds with 10 or less carbon atoms (monoterpenes and benzene derivatives), and oxygenated compounds with 15 or more carbon atoms (sesquiterpenes and lactones). The contribution of each compound family has been calculated as the sum of the area contribution of all compounds

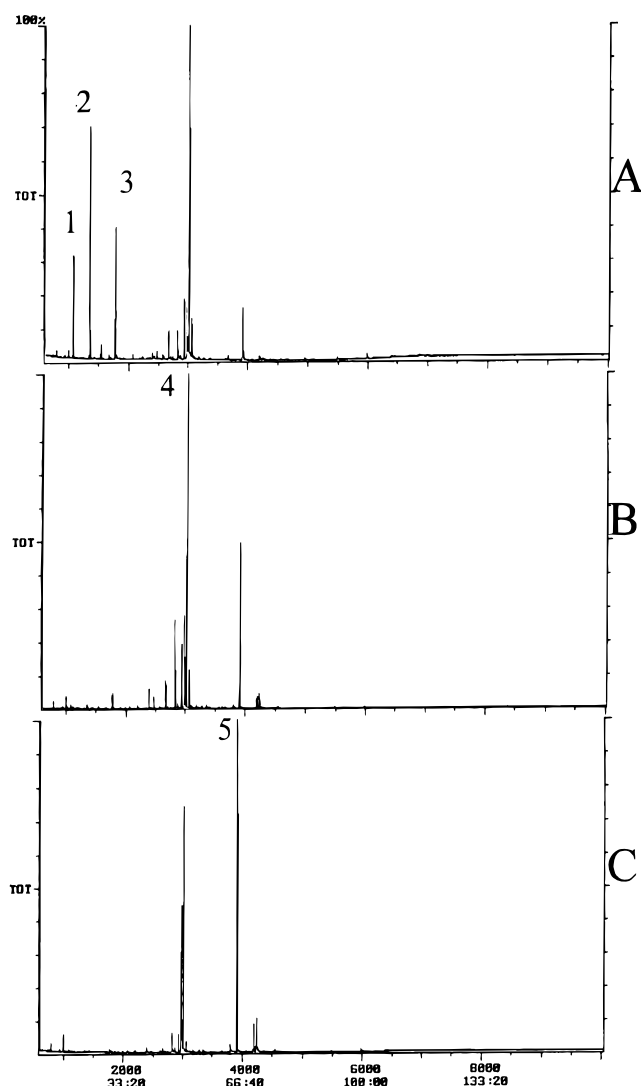


Figure 1. GC traces of three fractions extracted at 80 bar and 40 °C and collected in the second separator: (A) fraction recovered after 20 min of extraction; (B) fraction recovered between 360 and 480 min of extraction; (C) fraction recovered between 690 and 750 min of extraction. Peaks: 1, 1,8-cineole; 2, methyl benzoate; 3, methyl salicylate; 4, *trans*-methylisoeugenol; 5, eugenyl acetate.

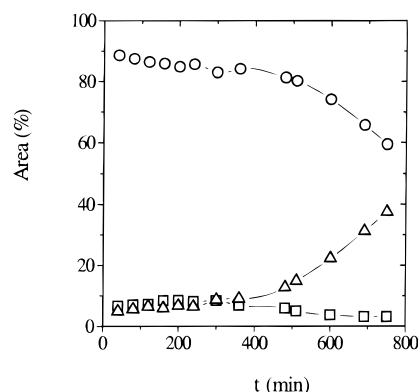


Figure 2. Area percentages versus the extraction time for compound families forming tuberoso volatile oil (test performed at 80 bar and 40 °C): □, hydrocarbon compounds; ○, oxygenated compounds ($\leq C_{10}$); △, oxygenated compounds ($\geq C_{15}$).

belonging to that family. Tuberoso oil contains a low percentage of hydrocarbon compounds (monoterpenes and sesquiterpenes) and their percentage decrease by increasing the extraction time. The percentage of oxygenated compounds with 10 or less carbon atoms

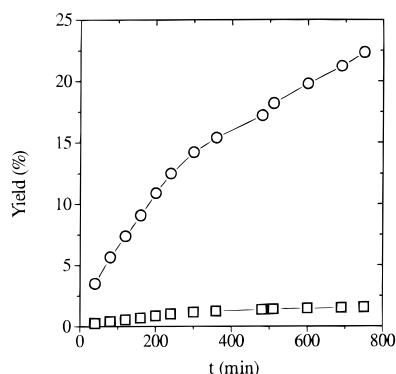


Figure 3. Volatile tuberoso oil yield (%) versus the extraction time (test performed at 80 bar and 40 °C): □, compounds not responsible for fragrance; ○, fragrance compounds.

also decreases during the extraction, whereas the percentage of oxygenated compounds with 15 or more carbon atoms increases especially at extraction times longer than 450 min. It means that different mass transfer resistances characterize the various compound families during the extraction process and the extraction time plays a relevant role on the final composition of tuberoso oil. Moreover, by interrupting the extraction of the oil at different times, it is possible to fractionate the fragrance and to obtain an extract in which top-notes or bottom-notes prevail.

In Figure 3, we reported the yield (%) of the different compound families against the extraction time. In this case we divided the tuberoso oil compounds in two groups: the fragrance compounds (oxygenated compounds) and the nonfragrance compounds (hydrocarbon compounds). The yield curves show an exponential trend against the extraction time: the hydrocarbon compounds one get flat after the first 300 min of extraction, whereas the yield curve of fragrance compounds asymptotizes only when the complete extraction is performed (after 750 min). Therefore, the contribution of hydrocarbon compounds to the oil is very low (yield of 1.6% by weight of the charged material) while the yield of fragrance compounds was measured to be 22.4% by weight of the charged material.

In Table 1, we also reported the detailed composition of waxes recovered in the first separator (waxes column) at the end of an exhaustive extraction test performed at 80 bar and 40 °C. This extract was solid, colorless, and odorless and was constituted mainly by *n*-pentacosane (30.7%), *n*-heptacosane (43.4%), and *n*-nonacosane (15.0%). Therefore, a good fractionation of the extract was obtained and small quantities of paraffins were recovered in the volatile oil (less than 0.9%).

The supercritical extract obtained at the optimum extraction and fractionation conditions was also compared to the absolute of tuberoso obtained by ethanol solubilization. The detailed identification of the SFE extract obtained by mixing together all the fraction recovered in the second separator during the whole extraction process at 80 bar and 40 °C was reported in Table 2 (SFE column). In the same table the detailed identification of the compounds constituting the "absolute" (absolute column) is also reported. In the SFE extract, methyl benzoate and methyl salicylate are present in percentages of 3.2% and 2.6%, respectively, whereas their percentages are 9.0% and 4.9%, respectively, in the ethanol extract. The overall percentage of the two lactones is nearly the same in the two extract (8.9% in SFE oil and 7.7% in the absolute), whereas the

Table 2. Comparison between the Volatile Oil Obtained by Supercritical Fluid Extraction at 80 bar and 40 °C (SFE Column) and the Product Obtained by Alcohol Solubilization (Absolute Column)

compound ^a	SFE oil (%) ^b	absolute (%) ^b
octen-1-ene	—	0.38
<i>n</i> -octane	—	0.25
benzaldehyde	0.10	—
β -pinene	0.11	0.14
<i>p</i> -cymene	0.16	—
1,8-cineole	1.03	7.94
methyl benzoate	3.21	8.98
2-methylbenzonitrile	tr ^c	0.55
benzyl acetate	tr	0.21
α -terpineol	0.87	0.70
methyl salicylate	2.56	4.92
ethyl benzoate	tr	0.42
phenyl butylacetate	tr	1.43
methyl anthranilate	tr	—
eugenol	0.22	1.08
β -bourbonene	0.09	0.20
methyleugenol	1.29	2.00
caryophyllene	0.12	0.23
β -gurjunene	tr	0.11
<i>cis</i> -isoeugenol	0.17	6.02
<i>cis</i> -methylisoeugenol	0.02	0.06
<i>cis</i> - β -farnesene	0.17	0.17
valencene	3.52	5.26
nepetalactone	6.59	6.58
dehydronepetalactone	2.33	1.10
<i>trans</i> -methylisoeugenol	60.68	31.55
α -farnesene	1.40	2.94
eugenyl acetate	15.33	12.97
benzyl benzoate	0.04	1.44
<i>n</i> -nonadecane	—	0.13
<i>n</i> -heicosane	—	0.16
<i>n</i> -heneicosane	—	0.30
<i>n</i> -pentacosane	—	1.80

^a The compounds are reported in elution order on a DB-5 column. ^b Area percentages are reported without correction factor. ^c tr < 0.01%.

content of *trans*-methylisoeugenol and eugenyl acetate is significantly larger in the SFE extract (60.7% versus 31.5% and 15.3% versus 12.9%, respectively) than in the absolute. A total of 2.38% of paraffins was detected in the ethanol extract even if it was submitted to many cooling cycles, spin-drying, and ultrafiltration. Due to these treatments a very low yield was obtained (11%) when compared with the supercritical extract yield (24%). Only traces of paraffins were detected in the SFE oil.

After the exhaustive extraction step at 80 bar and 40 °C, a second step of extraction was performed on the same charge by operating at 100 bar and 40 °C for 100 min. The scope of this second run was to extract the high molecular weight compounds present in tuberoso concrete. The high-pressure step gave in the second separator a product (yield of 11.8% by weight of the charge) that is still liquid but viscous, with a dark orange color and without the characteristic tuberoso aroma. The detailed identification of the compounds that formed this extract was reported in Table 1 (HP column). It consisted of some paraffins (14.8%) and a high percentage of long-chain hydrocarbon alcohols (among them *n*-tetracosan-1-ol (13.3%) and *n*-pentacosan-1-ol (8.4%) were the major compounds). Therefore, though non-negligible percentages of oxygenated compounds (30.8%), nepetalactone (13.5%), *trans*-methylisoeugenol (9.4%), and eugenyl acetate (18.6%) were detected, the tuberoso fragrance was largely modified.

The composition of this last extract confirms that the extraction pressure and the temperature used in the

first step of the process were opportunely chosen to avoid the coextraction of the long-chain alcohols that give to the extract an unpleasant fragrance (Fenaroli, 1963). Thus, by increasing the extraction pressure, it is possible to increase the yield of the process, but a less valuable tuberose oil is obtained.

ACKNOWLEDGMENT

The work was partly developed in the framework of Progetto Strategico "Tecnologie Chimiche Innovative", CNR, Rome, Italy.

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Received for review August 5, 1996. Revised manuscript received December 17, 1996. Accepted December 17, 1996.®

JF960589E

® Abstract published in *Advance ACS Abstracts*, February 1, 1997.