Major Volatile Constituents of Egyptian Rose Absolute

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Rose absolute *(Rosa gallica Linné)* is an expensive natural product which plays an important role in the manufacture of many famous luxury perfumes. The production of rose absolute in Egypt has increased in recent years.

The composition of rose absolute has not been throughly investigated except for a few reports.[1,8,11,12] However, the composition of rose oils from different origins has been studied in detail.[2,3,5-7,10,14,16]. This paper attempts to identify some of the major volatiles of rose absolute prepared from rose variety largely cultivated and processed in Egypt.

EXPERIMENTAL

Preparation of Rose Absolute

Rose absolute used in this study was prepared in the Research Department of Cairo Food Flavors and Essences Co., Cairo, Egypt. Rose flowers *(Rosa gallica var. aegyptiaca)* were extracted with a high-grade n-hexane to produce 0.29% of rose concrete. Rose absolute was then isolated from the concrete by extraction into a perfumery grade ethyl alcohol. Using essentially the technique described by Guenther,[4] the alcoholic extract was filtered and distilled under vacuum to obtain rose absolute (58.73%). The yield of absolute from rose flowers was 0.17%.

Isolation of Rose Volatiles

A 200 g sample of rose absolute was distilled under a vacuum of 0.1 mm Hg in all glass apparatus with a specially designed flask. The temperature of the distillation was maintained at 100°C for one hour. The condensate collected in a series of four cooled traps was washed with diethyl ether and then concentrated using a spinning band distillation apparatus (K-500 400,

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Kontes Glass Co., Vineland, New Jersey). The distillate obtained was considered as Rose Volatiles I.

The distillation was continued for another hour at 200°C. The condensate obtained was treated in a similar manner to obtain Rose Volatiles II. The total distillate of both Rose Volatiles I and II amounted to 41.7% of the starting rose absolute. The method used was essentially the same as that used previously for jasmine oil.[13]

Gas Chromatographic Fractionation of Rose Volatiles

A Perkin-Elmer Sigma 3 Gas Chromatograph with a flame ionization detector was used for the fractionation of rose volatiles.

A 12 ft x ⅛ in o.d. stainless steel column packed with 10% Carbowax 20M on 50/60 mesh Anakrom ABS was used for the first fractionation. The column temperature was programmed from 70° to 215°C at 5°/min (for Volatiles I), from 110°-215°C at 3°/min (for Volatiles II), and held at the upper limit. The flow rate was 30 ml/min. Under these operating conditions, Rose Volatiles I and II were separated into 18 and 21 broad fractions, respectively (see figs. 1 and 2). Each of the broad fractions was collected in "hairpin" traps made of 2 mm o.d. borosilicate glass.[15]

The major broad fractions (peaks 1, 7, 13, 14, 15, and 16 of Rose Volatiles I) and (peaks 1, 13, 14, 15, and 16 of Rose Volatiles II) were each rechromatographed using a 12 ft x ⅛ in. o.d. stainless steel column packed with 10% OV-101 on 60/80 mesh Chromosorb W. These major fractions represented about 98.01% and 94.71% of the isolated Rose Volatiles I and II, respectively.

Identification of Rose Volatiles

The major components of rose volatiles were identified by comparing their infrared and mass

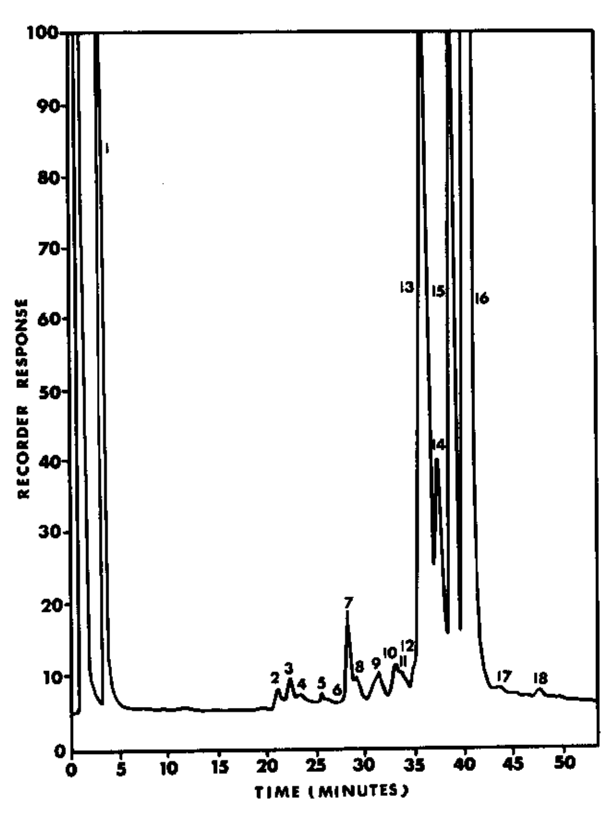

Figure 1. Gas chromatogram of distillate of Rose Absolute—Volatiles I.

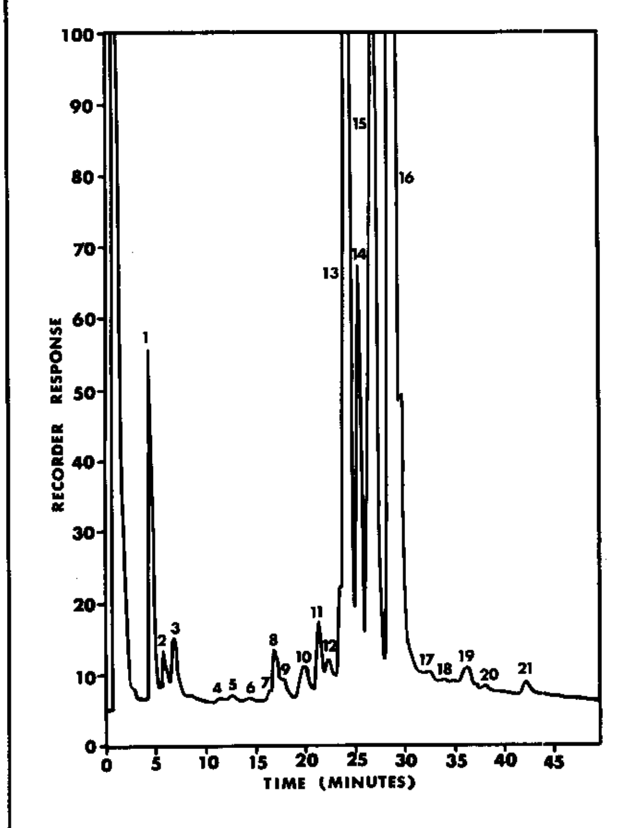

Figure 2. Gas chromatogram of distillate of rose absolute—Volatiles II.

Table I. Compounds Identified in the Major Volatiles of Rose Absolute

Peak Number(1)	Identified As	Relative % in Volatiles
Rose Volatiles I		
	Esters	
13-2-2	* Benzyl acetate	0.28
14-1-1	* Beta-Phenylethyl formate	0.19
15-4-2	Beta-Phenylethyl acetate	2.23
13-4-3	Citronellyl acetate	0.44
15-4-3	* Neryl propionate	0.92
15-6-2	* Geranyl isobutyrate	1.23
	Alcohols	
15-2-3	* Benzyl alcohol	1.38
16-3-1	Citronellol	75.23
Rose Volatiles II		
	Esters	
14-6-1	Neryl acetate	0.26
15-4-2	* Citronellyl propionate	0.04
	Alcohols	
16-1-1	2-Phenylethanol	84.32
15-4-3	Nerol	0.03
14-7-2	* 2,7,11-Trimethyldodeca-2,6,10-trien-1-ol	0.29
	Ethers	
14-7-3	* Digeranyl ether	0.20
	Hydrocarbons	
1-3-2	Beta-Pinene	0.73
14-8-4	* Beta-Farnesene	0.84

(1) The first, second and third numerals indicate the number of GC peaks during the first, second and third chromatography, respectively

* Compounds not previously identified in rose absolute

spectra obtained with the published spectra.

Infrared spectra were recorded on a Beckman Acculab 4 infrared spectrophotometer (Beckman Instruments, Inc., Mountainside, New Jersey. Cell thickness was 0.1 mm and the solvent used was spectroquality grade CCl_4.

Mass spectra were recorded on a Du Pont Model 21-490 mass spectrometer with a jet separator interfaced with a Varian Moduline 2700 gas chromatograph equipped with a flame ionization detector. A 12 ft x ⅛ in o.d. stainless steel column packed with 3% OV-17 on 70/80 mesh Anakrom ABS was used. The ionization voltage was 70 V and the ion source temperature was at 230°C. Peak areas were measured by the integrator of a Hewlett-Packard GC Model 484OA.

RESULTS AND DISCUSSION

The isolated Rose Volatiles (I and II) from rose absolute (41.73%) were first fractionated by preparative gas chromatography into 18 and 21 fractions, respectively (figs. 1 and 2). The major fractions were collected and then rechromatographed on a second column so that pure subfractions could be collected for identification. Table I lists the compounds identified in the major volatiles of rose absolute.

Alcohols are by far the major constituents in the volatiles of Egyptian rose absolute amounting to 76.61% and 84.64% of Rose Volatiles I and II, respectively. Among the five identified alcohols, citronellol (75.23% of Rose Volatiles I) and 2-phenylethanol (84.32% of Rose Volatiles

II) were predominant. Such large amounts of both alcohols present appear to contribute significantly to the aroma of Egyptian rose absolute. Citronellol and 2-phenylethanol were previously identified in Bulgarian rose absolute by Nikolov et al.[11] However, benzyl alchol and 2,7,11-trimethyldodeca-2,6,10-trien-1-ol have not been reported before.

Esters are the second large group of compounds identified in the major volatiles of Egyptian rose absolute. Among the eight identified esters, β-phenylethyl acetate has been found in Hamanasu rose absolute.[12] It was found in a somewhat larger concentration (2.23% of Rose Volatiles I) in Eqyptian rose absolute. Five of the esters identified have not been reported previously.

Another new compound found in rose absolute was digeranyl ether which was the only ether identified in the present study.

All of the identified compounds in this study constituted approximately 81.9% and 86.7% of the Rose Absolute Volatiles I and II, respectively.

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