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Danielle Barth, Marie Debacq, Alain Brembilla, Dominique Petitjean, Michel Dirand. Supercritical carbon dioxide purification of n-octacosane obtained by chemical synthesis. 5th Meeting on Supercritical Fluids, Mar 1998, Nice, France. pp.1005-1010. hal-01500251

HAL Id: hal-01500251

<https://hal.science/hal-01500251>

Submitted on 12 Apr 2017

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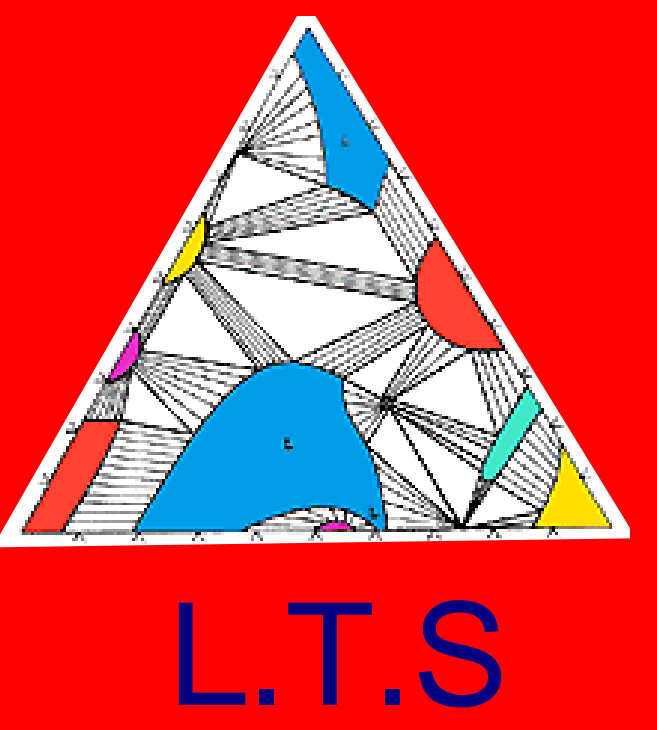


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SUPERCRITICAL CARBON DIOXIDE PURIFICATION OF *n*-OCTACOSANE OBTAINED BY CHEMICAL SYNTHESIS

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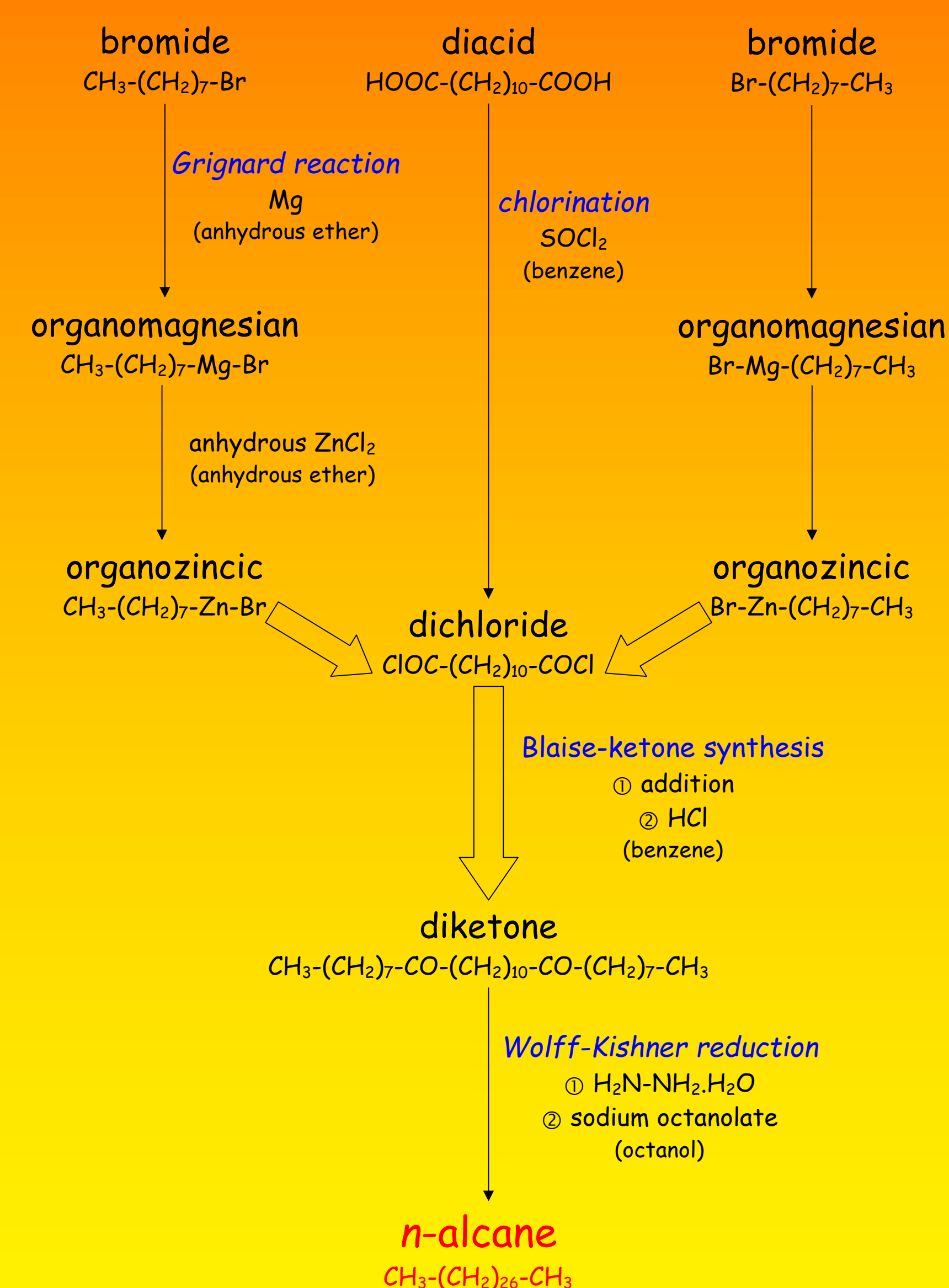
INTRODUCTION

The evaluation of the crystallization point of fuels can be only accessed from the thermodynamic properties knowledge of the higher *n*-alkanes which compose them, and if any, whose crystalline structures have been solved. An equimolecular binary mixture *n*-octacosane / *n*-hexacosane should be a good model for the study of the complex paraffinic deposits which form in the oily fluids. To this end, it is essential to have at one's disposal, the highest purity material. The commercial products contain, as main impurities, branched-chain and/or linear chains of closely related homologues which are extremely difficult to eliminate.

The purpose of this work deals with the synthesis of one component of the binary mixture (*n*-octacosane) and its supercritical carbon dioxide purification. The purity of *n*-octacosane obtained was tested by several ways (melting point, gas chromatography, calorimetric analysis, X ray diffraction) and compared with the commercial product.

CHEMICAL SYNTHESIS

REACTION SCHEME



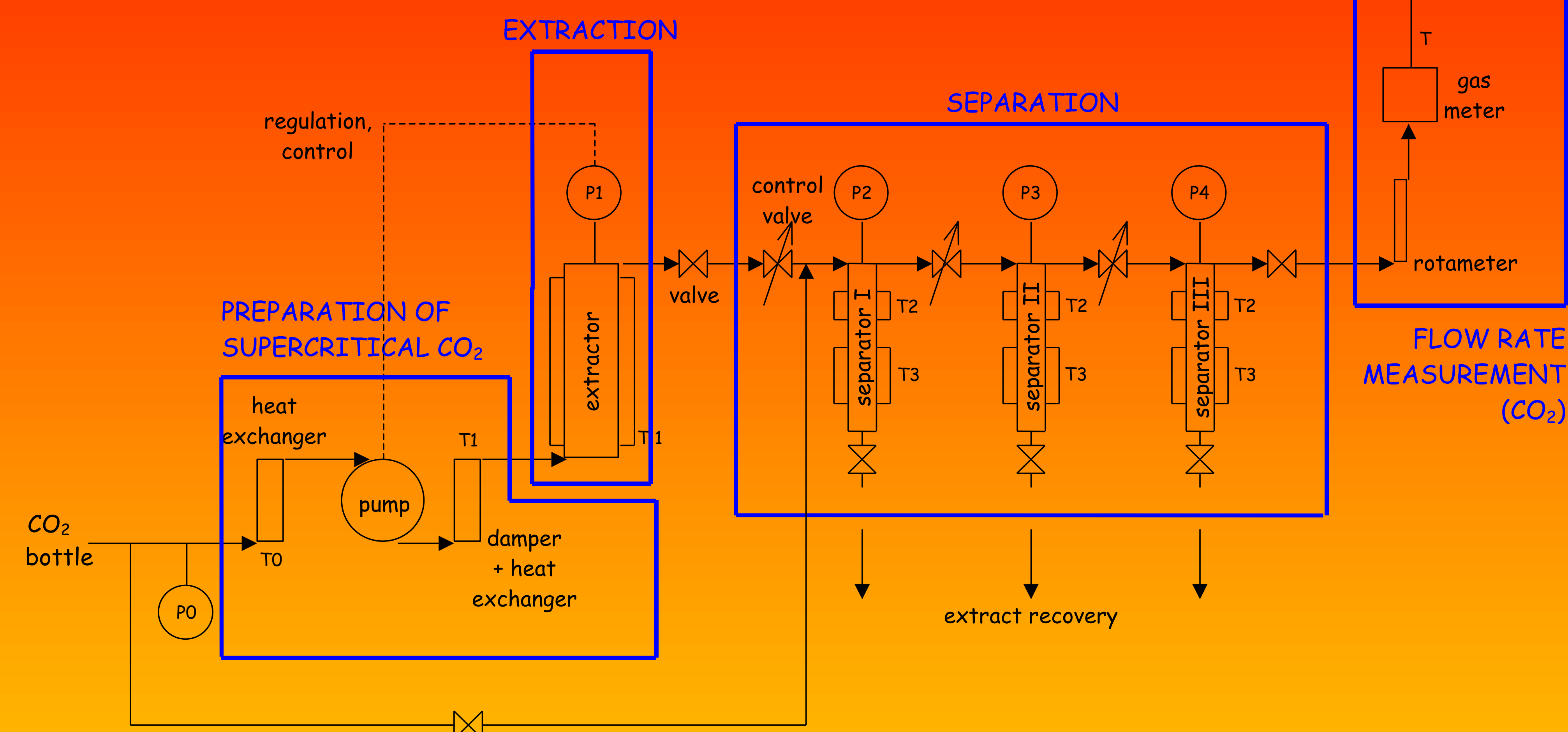
CHARACTERIZATION

melting point
58 °C
infrared spectrum
CH₃, CH₂ groups
+ CO group
¹³C RMN spectrum
28 carbons

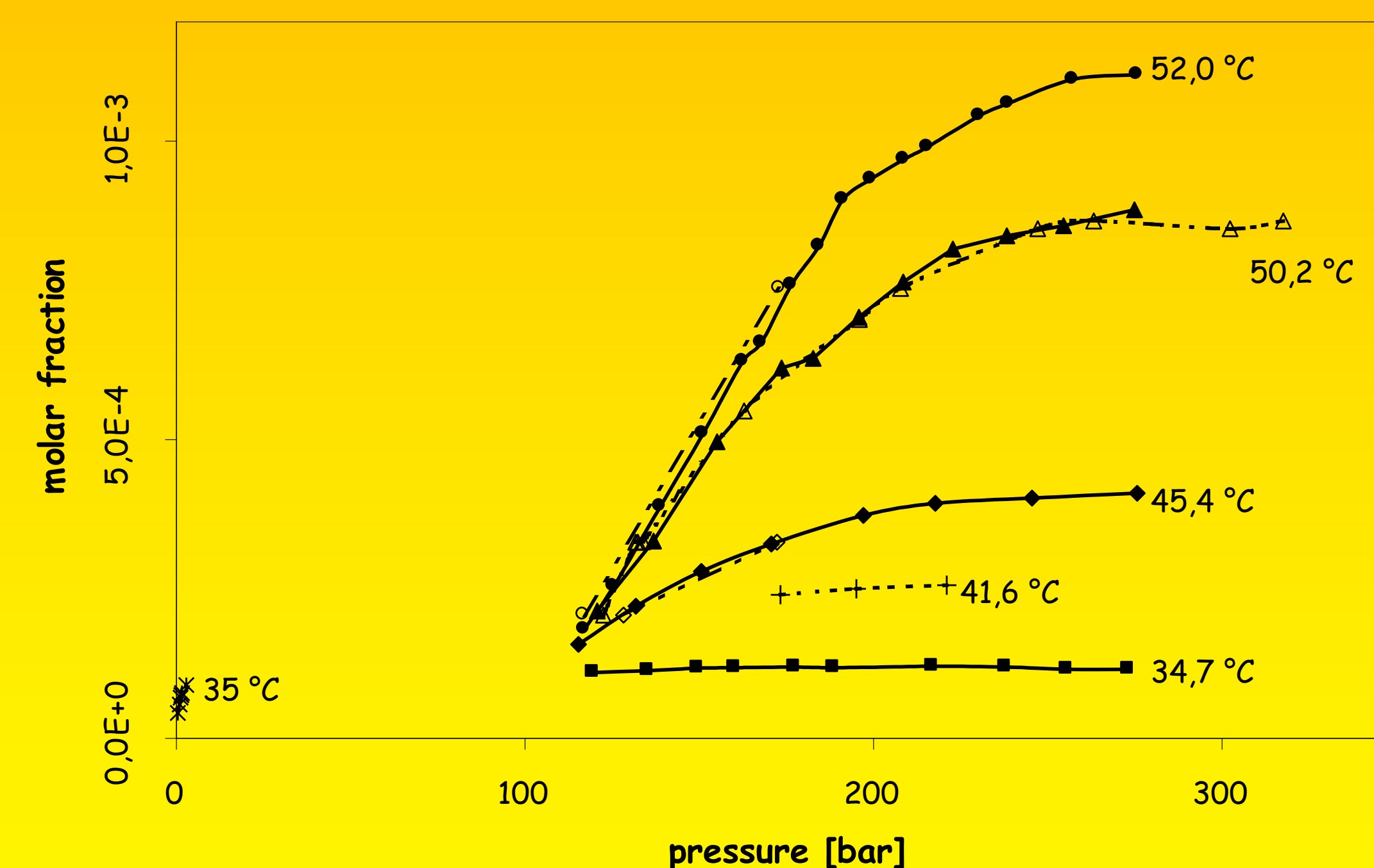
structural analysis
alkane rays
+ diketone rays
gas chromatographic analysis
alkane pic
+ diketone pic

synthetized product = *n*-octacosane + diketone

EXTRACTION PLANT



SOLUBILITY



[1] McHUGH, SECKER, YOGANT - J. Ind. Eng. Chem. Fundam. - vol. 17 - n°1 - 1984
[3] GRISCIK, ROUSSEAU, TEJA - J. of Crystal Growth - vol.155 - 1995
[4] SMITH, CAMBELL, ROBERTS, SHERWOOD - J. Mater. Chem. - vol.4 - n°6 - 1994

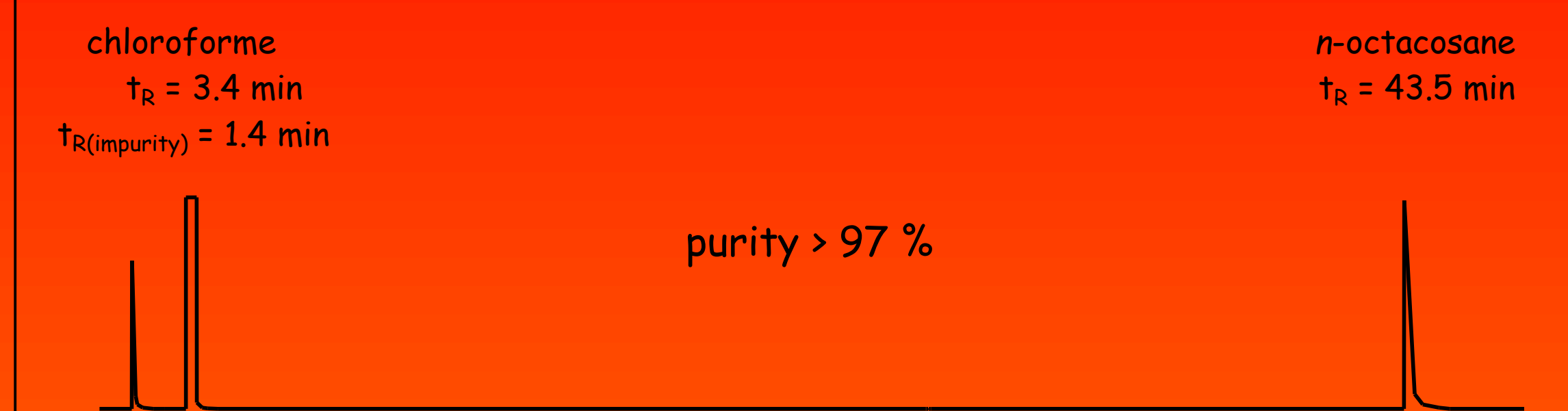
EXTRACTION RESULTS

exp.	product used	mass feed [mg]	T [°C]		P [bar]				time [min]	mass in sep. I [mg]
			extract. T1	sep. T2=T3	extract. P1	sep. I P2	sep. II P3	sep. III P4		
1	C28 Fluka 98 %	620.8	45	60	110				75	2.3
3	C28 Fluka 98 %	107.9	45	40	110	50 to 60	16 to 24	1 to 5	60	0.6
4	89.5 % mas C28 + 10.5 % diketone	101.0	45	40	110					3.8
5	89.8 % mas C28 + 10.2 % diketone	101.3	45	40	110	40 to 50	10 to 15	1 to 3	90	22.2
6	88.3 % mas C28 + 11.7 % diketone	168.6	55	40	110	50 to 60	10 to 20	1 to 2	90	7.0
7	synthetised octacosane	102.6	55	40	110	65 to 70	20 to 30	about 2	75	22.0
8	synthetised octacosane	154.4	55	40	110	65 to 75	20 to 30	3 to 5	50	7.9
9	C28 Fluka 98 %	121.8	55	40	110				70	3.7

CHARACTERIZATION OF EXTRACT

The extract has a very nice appearance (a very fine powder, sparkle and white) in accordance with the usual appearance.

gas chromatographic analysis



thermal analysis

Both extract and commercial products have solid-solid transition δ_0 (monoclinic phase) to α -RII (rhombohedral Rotator phase). The melting points are similar.

	solid-solid transition	melting point
extract	56.3 °C	60.6 °C
commercial product	56.7 °C	60.5 °C

structural analysis (at room temperature)

X-ray diffraction experiment (counter diffractometer, copper radiation λ , K α Cu) shows that the structure of the two samples (extract and commercial) are identical. The average interplanar distance for the extract is in good agreement (3.37 nm) with that indicates in the litterature (3.34 nm).

angle [°K α Cu]	1.25	2.57	3.90	5.25	6.57	7.90
distance	3.53	1.71	1.13	0.84	0.67	0.56
00l ray	001	002	003	004	005	006

[6] GERSON, ROBERTS, SHERWOOD, Acta Cryst., B47, 1991

extract = *n*-octacosane

CONCLUSION

The good purity of the extract was established by gas chromatographic, thermal and structural analysis, and compared with the commercial product. The purity of our *n*-octacosane is comparable to the Aldrich one's (99 %), and our product is free from branched-chains and homologues.

Future prospects :

We try to optimize the chemical synthesis in order to improve the yield.
We also try to optimize the withdrawn of the solute in order to improve the mass balance.
Finally, the structural analysis at different temperatures is in progress.