Ertec, 2-Theta, March 2009

Microwave Digestion of Samples

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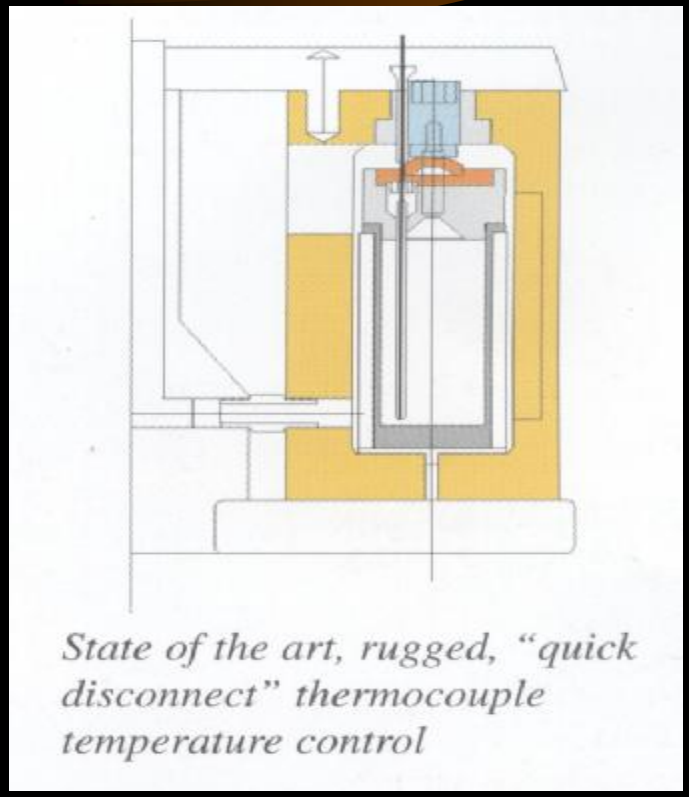
Wroclaw, Tesin 2009

High pressure MW systems

Ethos plus
from Milestone

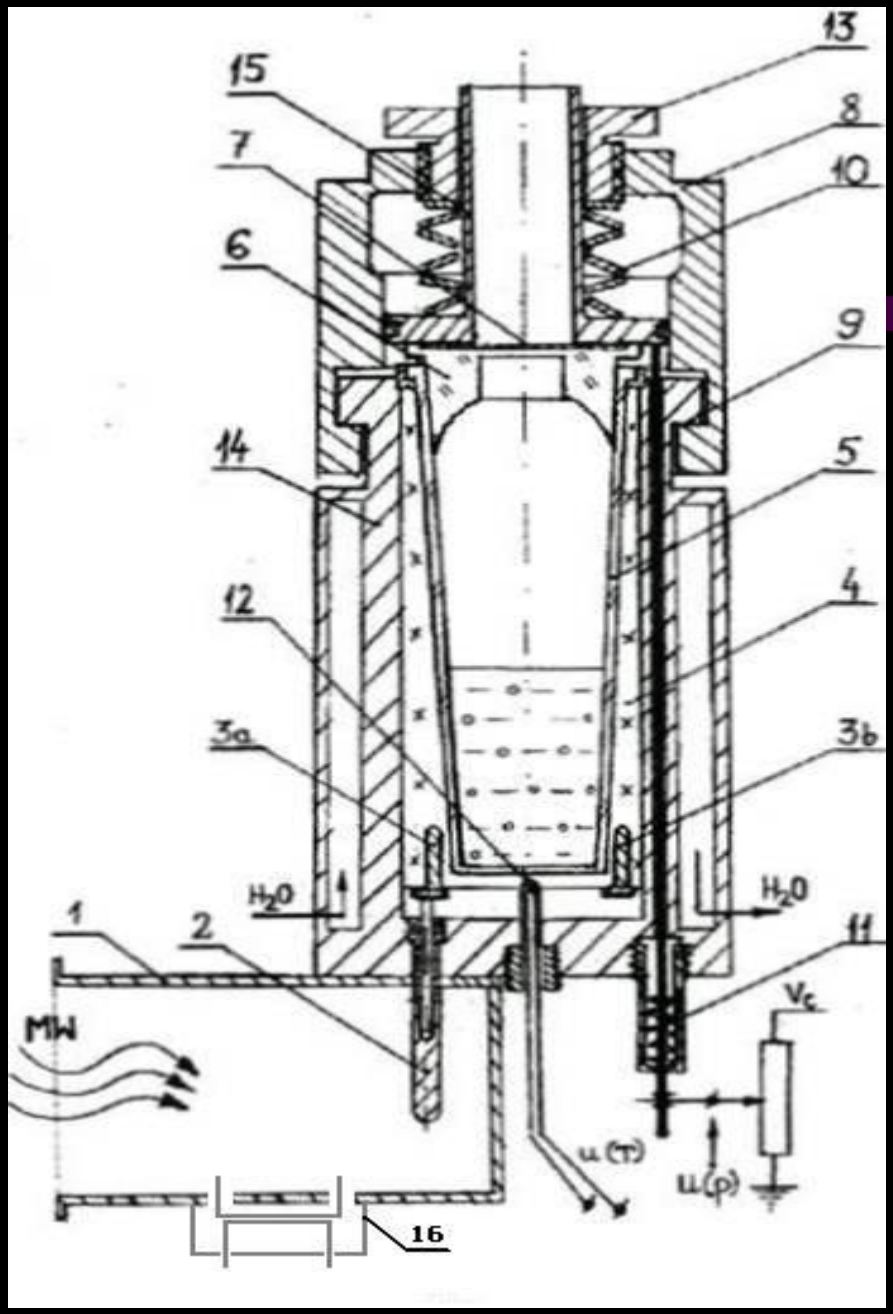


digestion vessel
Ethos

High pressure digestion system with water cooled steel jacket

Ertec console

Reactor/mineralizer Ertec



Cross-section of Magnum digestion vessel

Recovery at microwave and conventional dissolution of ceramics

Table7. Dissolution speed of „conventional” and microwave closed approaches			
	Conventional Teflon bomb		Microwave digestor
Matrix	(h)	(min)	(min)
Al_2O_3	24	1440	20
AlN	2	120	15
BN	5	300	15
Si_3N_4	6	360	15

Table 8. Analytical results ($\mu g\ g^{-1}$) for determination of impurities in commercial ceramic powders obtained by "conventional" closed-vessel and microwave decomposition and analysis by MIP-AES

Sample	Method	Al	Ca	Cu	Fe	Mg	Na	Zn
Al_2O_3, Aldrich	conventional		5	8	37	12	210	140
	microwave		7	9	39	10	220	126
AlN, Aldrich	conventional		23	16	145	12	120	48
	microwave		25	20	150	13	128	45
BN, Aldrich	conventional	100	130	20	50	30	60	83
	microwave	110	125	24	50	25	65	78
BN, Merck	conventional	142	110	30	96	41	99	110
	microwave	140	105	29	100	44	104	111
Si_3N_4 (LC 12-S), H. C. Starck	conventional	310	41	12	115	31	54	21
	microwave	320	40	10	118	30	55	20
	accepted value[a]	300	40		120			
Si_3N_4 (LC 12), H. C. Starck	conventional	410	50	9	630	42	49	30
	microwave	405	50	10	610	45	50	33
	accepted value[a]	400	50		600			
Si_3N_4, Aldrich	conventional	12	25	20	195	7	38	10
	microwave	11	24	22	205	8	40	12
	accepted value[a]	10			200	5		

[a] Results are those reported by the manufacturers.

TOC

Table I Total residual carbon content in High pressure focused microwave heated digested samples of Bovine Liver (NIST–SRM 1577a)

Digestion method	Sample mass (g)	Final volume (ml)	Digestion time (min)	Residual carbon[a] in digestate ($\mu g ml^{-1}$)	Residual carbon[a] in dry sample ($mg g^{-1}$)	Efficiency of oxidation[b] (%)
Microwave heated bomb	0.1	10	4	30 ± 3	3 ± 0.3	99.4
High Pressure Asher	0.1	10	120	20 ± 2	2 ± 0.2	99.6

[a] Total carbon content of undigested sample: 510 ± 10 $mg g^{-1}$ (n=3), dry mass basis. Mean and standard deviation reported
[b] Five measurements from a triplicate sample preparation

Table II Total residual carbon in High pressure focused microwave heated digested samples[a] (Triplicate measurements from a duplicate sample preparation. Mean and standard deviation reported)

Material	Original carbon content[b] (%)	Fat (%)	Lipids (%)	Residual carbon content[c] (%)	Residual carbon[f] (%)	Efficiency of oxidation (%)
NRCC TORT-1	42.5 ± 0.9	d	d	0.1	0.4	99.6 ± 2.0
NRCC DORM-1	44.4 ± 0.9	5	d	0.2	0.9	99.1 ± 2.0
NIST 566a	49.1 ± 1.0	d	d	0.2	0.4	99.6 ± 2.0
NIST 577	49.5 ± 1.0	d	d	0.3	0.6	99.4 ± 2.0
NRCC DOLT-2	50.6 ± 1.0	20	d	0.4	0.8	99.2 ± 2.0
NRCC LUTS-1	38.8 ± 1.2[e]	d	55[e]	0.6[e]	1.6[e]	99.0 ± 2.0

[a] Digestion for 5 min at 100 W
[b] Total carbon content of undigested samples
[c] Dry mass basis
[d] No declaration

Digestion reagents

Solution of reagents	Matrix	Uwagi
HNO_3	Biological materials	incompletye digestion at atrmospheric pressure
$HNO_3 + H_2O_2$	Biological materials	Effective digestion of small samples
$HNO_3 + H2SO_4$	general use	Frequently used but promotes losses of easili volatile elements such as As, Ge, Hg, Se
$HNO_3 + HCl$	general use	aqua regia
$HNO_3 + HClO_3$	Biological materials	High oxidation potential, low loss of As, Hg, Se, In and so on .., safe
$HNO_3 + HClO_4$	Biological materials	Effective decomposition , low loss of Pb
$HNO_3 + HCl + HF$	general use	-
$HNO_3 + HF$	general use	-
$HNO_3 + HClO_4 + H2SO_4$	general use	Temperature regime must be controlled , loss of As, Hg, Fe Sb
HF	innorganic materials	-
$H2SO_4 + HClO_4$	general use	Small samples , denger of explosion
$H2SO_4 + H_2O_2$	general use	Losses of As, Hg, Ge, Ru, Se and many other volatile elements
$HClO_4$	Biological materials	Strong oxidizer, denger of explosion
$H_2O_2 + Fe^{3+}$	Biological materials except oils, fats and grees	Decomposition with OH^* radicals, low temperature of decomposition, good for large samples, no loss of elements

Useful Glassware Accessories

A glance at the new system from the front

Dwójka


EATEC
MW
MAGNUM II
EATEC
MW
MAGNUM II
EATEC
MW
MAGNUM II
EATEC
MW
MAGNUM II

Czwórka

Digestion of difficult organic samples with dry mass over 1g

ERTEC

Sample	Mass [g]	Reagents [ml]	Digestion procedure power [%] pressure set. [at]	Pressure reached (oscillation) [at]	Total digestion time [min]
Green coffee	1,5	6 HNO3	5'/60% 20-17at; 5'/100% 45-42at 10'/0%	45 (45-42)	20
Tea fix	1,5	6 HNO3, 2 H2O	5'/60% 20-17at; 5'/80% 45-42at; 10'/100%	45 (45-42)	20
Rice	2,5	8 HNO3, 4 H2O	10'/60% 20-17at; 10'/0% 45-42at; 10'/0%	105 (45-42)	30
Goulash soup	1,5	8 HNO3, 4 H2O	10'/60% 20-17at; 10'/100% 45-42at; 10'/0%	74 (45-42)	30
Dry mushrooms	2	8 HNO3, 4 H2O	10'/60% 20-17at; 10'/100% 45-42at; 10'/0%	45 (45-42)	30
Peanut	1,5	8 HNO3, 4 H2O	10'/60% 20-17at; 10'/100% 45-42at; 10'/0%	45 (45-42)	30
Poppy seed	2	8 HNO3, 4 H2O	10'/60%; 10'/100%; 10'/0%	82 (45-42)	30

Rodzaj próbki	Masa próbki	Procedura mineralizacji	Ciśnienie (max-min)	Rozpuszczalniki
Krew	0.5g	7min/100%,	45-42 at	7ml HNO_3,
Nasiona lnu	0.5g	7min/100%,	45-42 at	6ml HNO_3
Kłącze tataraku	0.5g	7min/100%,	45-42at	6ml HNO_3
Morszczyn	0.5g	7min/100%,	45-42 at	6ml HNO_3, 2ml H_2O_2
Włosy dziecka	1.0g	Krok 1) 1min/50%, 30sek/0%, Krok 2) 1min/60%, 30sek/0%, Krok 3) 2min/70%, 30sek/0%, Krok 4) 2min/80%,	20-17 at 30-27 at 35-32 at 45-42 at	7ml HNO_3, 3ml H_2O
Kopyto końskie	0.2g	15min/100%,	45-42 at	6ml HC1, 3ml H2 O_2
Kości wołowe	0.2g	25min/100%,	45-42 at	6ml HC1, 3ml H_2 O_2
Woda, ścieki	50ml	15/100%	45-42 at	7ml HNO
Wołowina chuda, świeża	5.0g	Krok 1) 2min/80%, Krok 2) 1min/60%, 30sek/0%, Krok 3) 2min/70%, 30sek/0%, Krok 4) 2min/80%,	20-17 at 30-27 at 35-32 at 45-42 at	10ml HNO_3
Kiełbasa średnio tłusta	1.0g	6min/100%,	45-42 at	6ml HNO_3
Ser twaróg	1.0g	10min/100%,	45-42 at	6ml HNO_3
Ser żółty	1.0g	10min/100%,	20-17	6ml HNO_3
Masło	1.0g	2min/60%, 2min/70%, 2min/80%, 5min/90%,	20-17 at 30-27 at 35-32 at 45-42 at	6ml HNO_3
Słonina	1.0g	10min/100%,	45-42 at	6ml HNO_3
Kości zwierzęce	0.2g	15min/100%,	45-42 at	6ml HC1, 3ml H_2O_2

PRÓBKI WYMAGAJĄCE SZCZEGÓLNEGO PODEJŚCIA

TABLETKI POWLEKANE
POLIMERY
PRÓBKI „NIESPODZIANKI” – OLEJ „TRANSFORMATOROWY”

GRAFIT Z ELEKTROWNI JĄDROWEJ MOCHOWCE – SŁOWACJA

Masa próbki – 0,1g
Odczynniki : 5 ml Perchloric acid 70 % + 2 ml H2O

Procedura rozkładu:

Krok 1 – Czas 5 min, Moc: 60 %, Pmax 20 atm, Pmin 17 atm, Tmax 300 ° C Tmin 295 ° C

Krok 2 – Czas 5 min, Moc: 80 %, Pmax 30 atm, Pmin 27 atm, Tmax 300 ° C Tmin 295 ° C

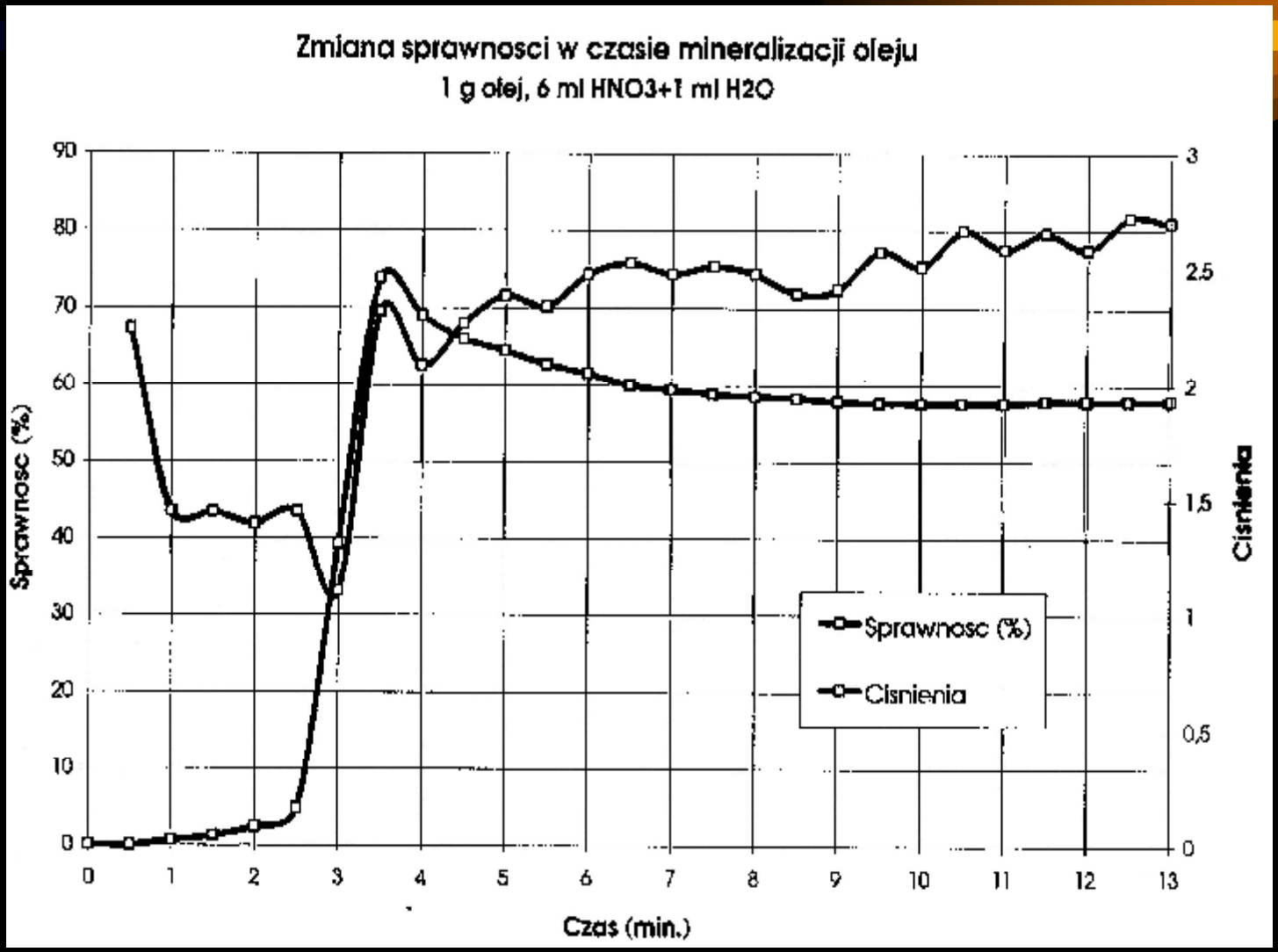
Krok 3 – Czas 10 min, Moc: 100 %, Pmax 45 atm, Pmin 42 atm, Tmax 300 ° C Tmin 295 ° C

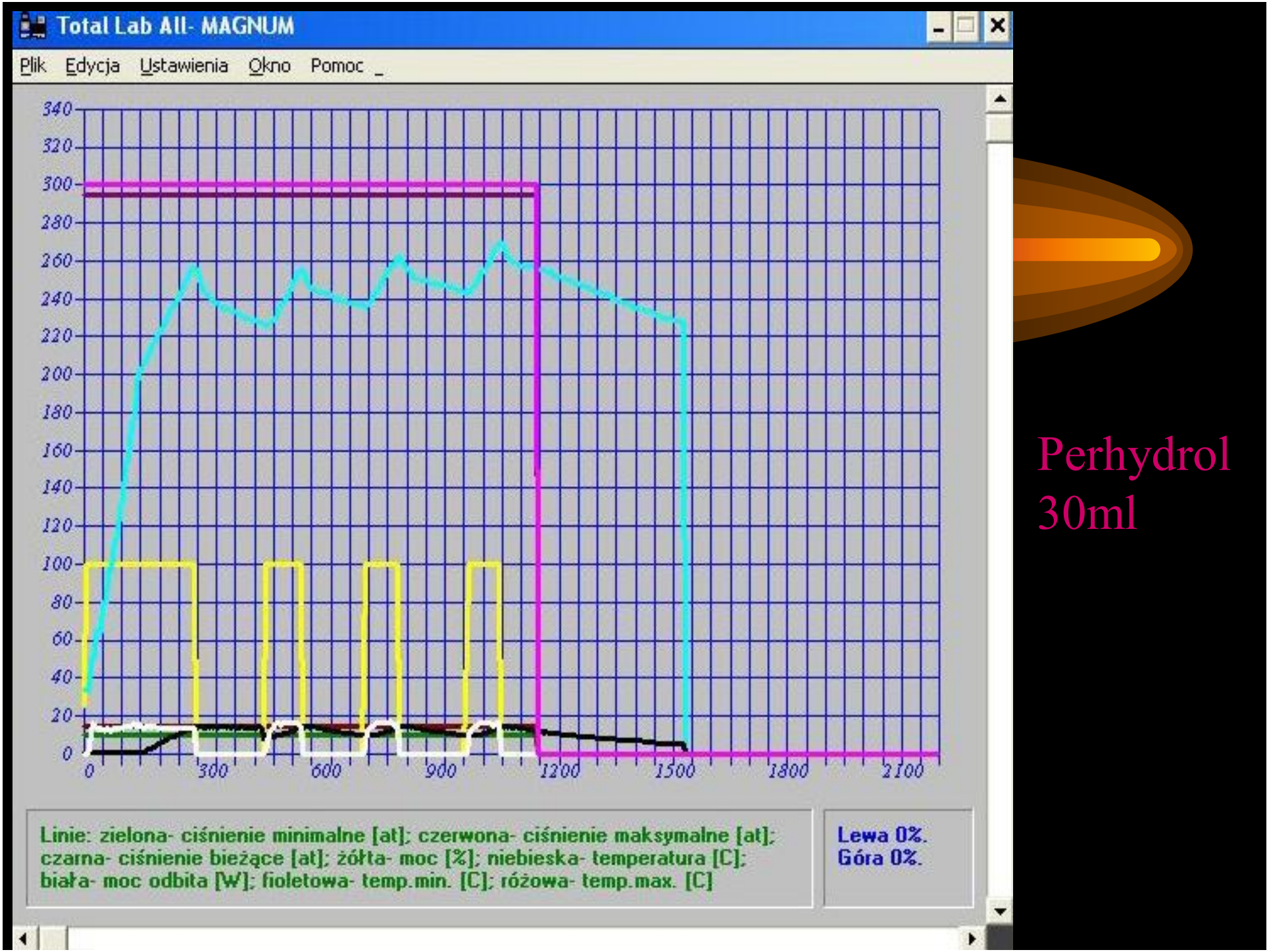
Bardzo dobry klarowny roztwór

Capability of different digestion systems

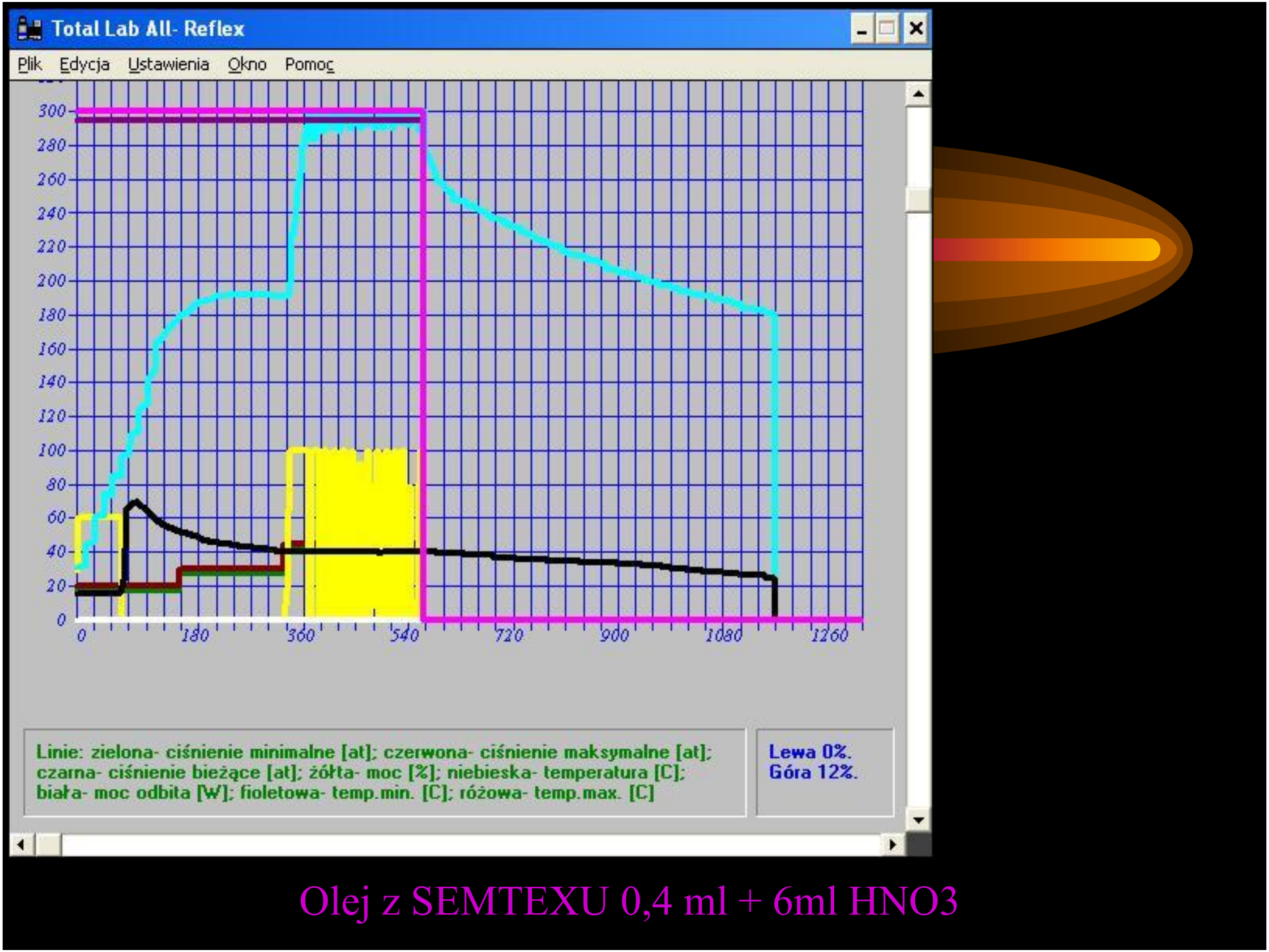
Kind of a Sample	Dry mass[g] (up to)	System ERTEC 2 modules Magnum (samples/day)	Microwave furnaces with 6 vessel carouselle (samples/day)
Organic	0.5 g	65	50
	1.0 g	50	sometimes impossible
	2.0 g	25	impossible!!
	5.0 g	12	impossible!!!
Mixed	0.5 g	50	36
	1.0 g	38	24
	2.0 g	19	usually impossible
	5.0 g	11	impossible!!
Non-organic (dissolution only)	0.5 g	35	30
	1.0 g	25	18
	2.0 g	13	sometimes impossible
	5.0 g	6	impossible!!

Power efficiency as an and point detection of digestion procedures





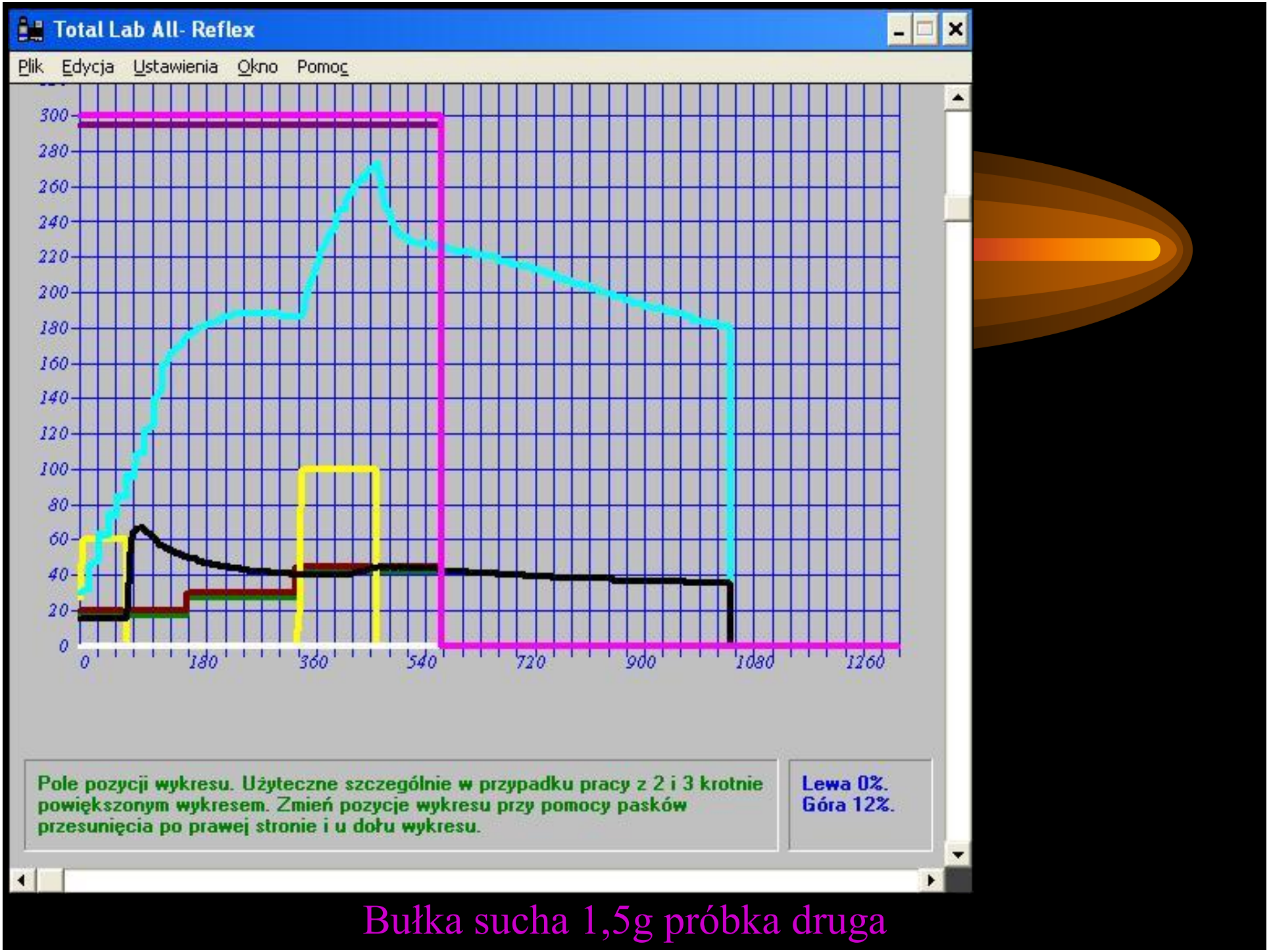
Perhydrol
30ml



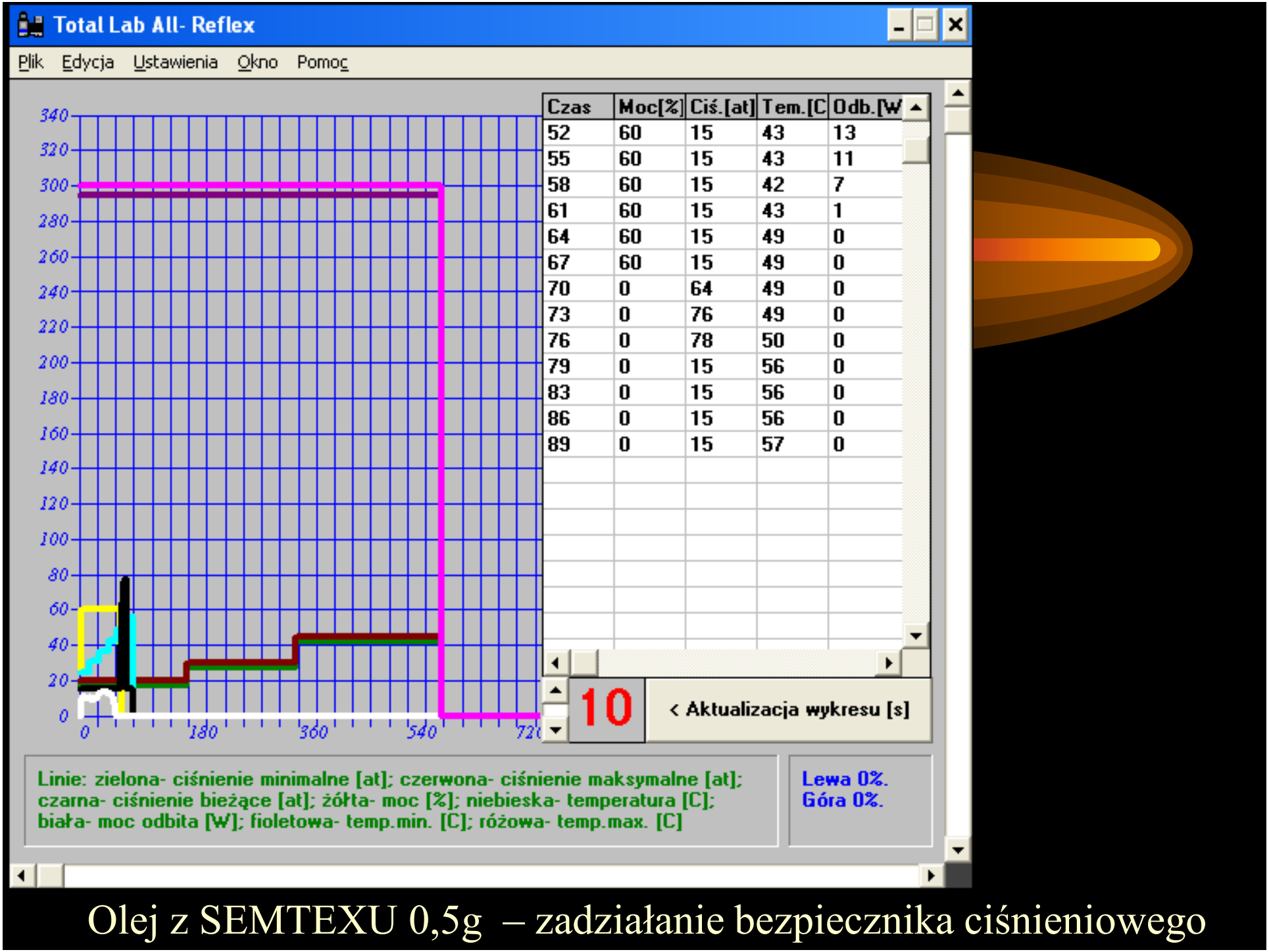
Olej z SEMTEXU 0,4 ml + 6ml HNO3

Temperature control –A new low - temperature Kjeldahl procedure

In this study, a microwave digestion system was used in place of the conventional block heating apparatus, typically used in the *Standard Methods*.[1] The sample size was chosen based on the difference in the solids content. The samples for standard solutions and sewage wastewaters, 5 mL of sample in the microwave digestion cell, was mixed with 5 mL concentrated H_2SO_4 and a digestion mixture consisting of 0.6 grams of K_2SO_4, and 0.02 gram of HgO. For aerobic sludge, 1 mL of sample was mixed with 10 mL of concentrated H_2SO_4 and the digestion mixture. Samples were subjected to microwave treatment with a temperature ramp from 23°C to 200°C for 10 min, followed by maintaining at 200°C for 15 min. It should be noted that the microwave digestion temperature was set in a much lower region than the conventional thermal digestion (380°C).



Bułka sucha 1,5g próbka druga



Olej z SEMTEXU 0,5g – zadziałanie bezpiecznika ciśnieniowego

Advantages of the new digestion system

1. A new more efficient coupling between the magnetron and the digestion vessel
2. Built-in reflectometer enabling measurements of microwave power reflected from the sample
3. Indication of the ‘well closed” digestion vessel’s head
4. Reduced more ergonomic hight of the digestor
5. A new user-friendly software with more useful data

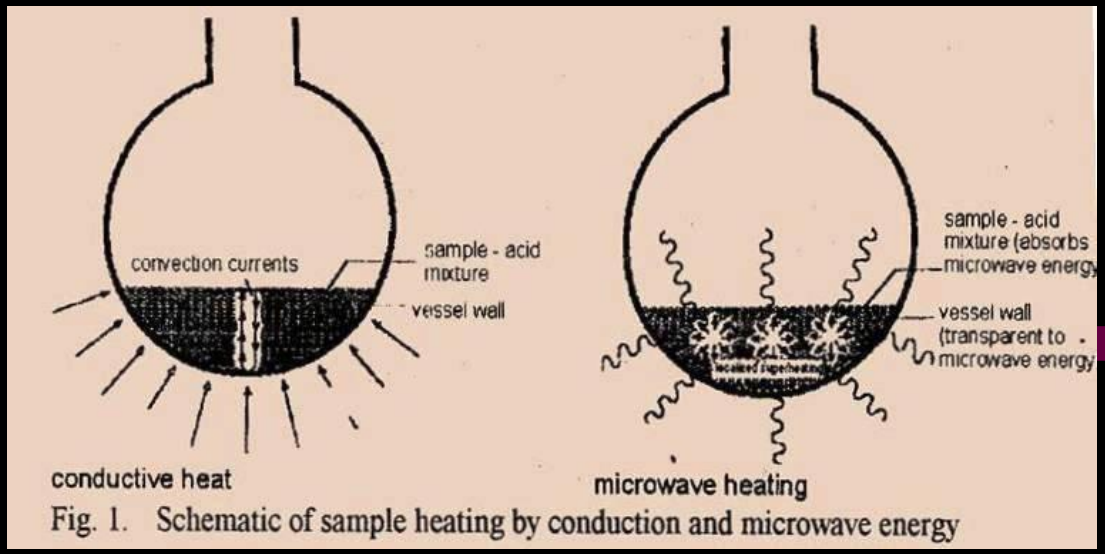


Fig. 1. Schematic of sample heating by conduction and microwave energy

Thank you for your attention