INTERNATIONAL ATOMIC ENERGY AGENCY
UNITED NATIONS EDUCATIONAL, SCIENTIFIC AND CULTURAL ORGANIZATION

INTERNATIONAL CENTRE FOR THEORETICAL PHYSICS
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SMR/348 - 19

EXPERIMENTAL WORKSHOP ON HIGH TEMPERATURE SUPERCONDUCTORS (11 - 22 April 1988)

OUTLINE OF EXPERIMENTAL ACTIVITIES

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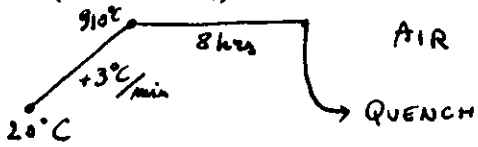
1) Preparation

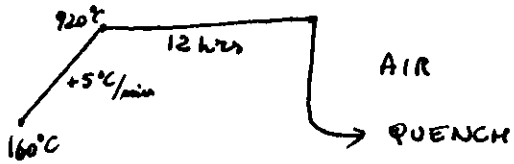
1.1. $YBa_2Cu_3O_{7-\partial}$ - Ceramic method (Yor Er)

Starting materials: $1/2(Y_2O_3)$or(Er_2O_3) 99.99%
BaO_2 99.6%
CuO 99.999%

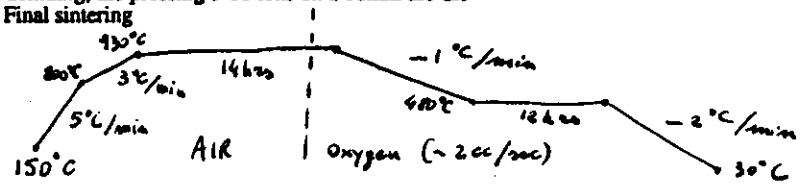
Mixing and grinding of appropriate amounts (cation ratios 1:2:3)
Firing of powders on an Al_2O_3 boat



1st diffraction sample
Grinding
Intermediate firing of powder



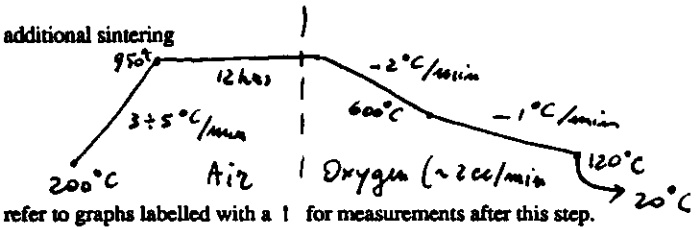
Grinding, die pressing 3-10 tons on a 16mm die-die
Final sintering



refer to graphs labelled with a * for measurements after this step

grinding, die pressing (4 tons) on a 16 mm die

additional sintering



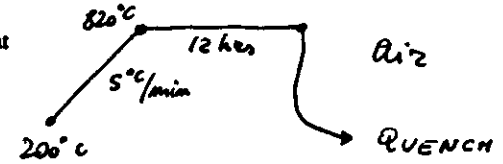
refer to graphs labelled with a ! for measurements after this step.

1.2. $Bi_2CaSr_2Cu_2O_{8+\partial}$

Starting materials 1/2 (Bi_2O_3) 99.9%
$CaCO_3$ 99.9%
$SrCO_3$ 99.995%
CuO 99.999%

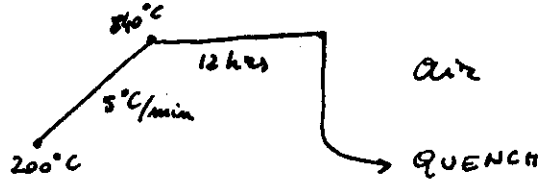
Mixing and grinding of the 1:1:1:2 ratio mixture of powders

Firing of powder in an Al_2O_3 boat



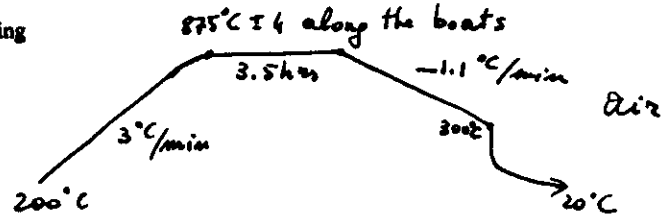
Grinding

Intermediate firing



Grinding, die pressing .5 tons on a 16mm die

final sintering



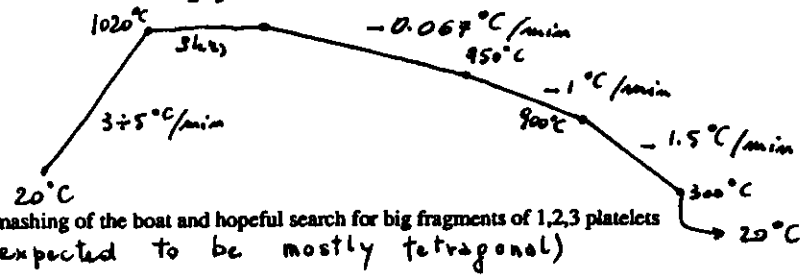
refer to graphs labelled with a Bi for measurments

1.3. $YBa_2Cu_3O_{7-\partial}$ single crystals

Starting materials: the same as 1.1.

Mixing and grinding of the 2:28:70 molar ratios of the powders
(in weight 2.2582 : 47.41 : 55.66)

Heat treatment in an Al_2O_3 boat



Smashing of the boat and hopeful search for big fragments of 1,2,3 platelets
(expected to be mostly tetragonal)

2) Measurements

2.1 Density: Rough measurements of pellet diameter, height and weight; D=P/V ± 10% Visual inspection under microscope of porosity, grain size, amount of green phase. RESULTS ON TABLES 1 & 2

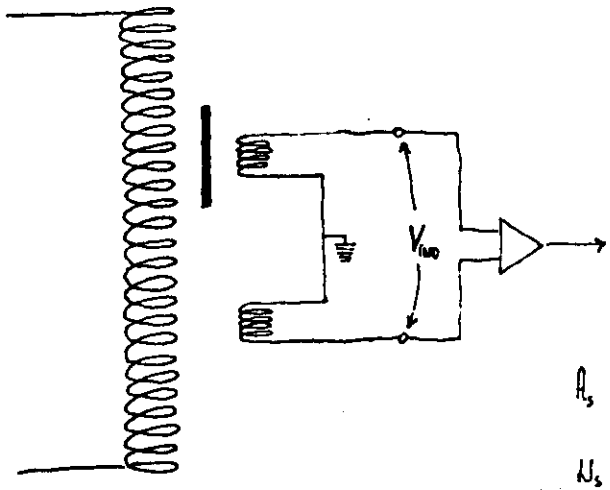
2.2. Levitation: Check of the levitation properties of LN_2 cooled pellets floating on $SmCo_5$ magnets.

2.3. a.c. Susceptibility
Scheme fig. A
Schetch of cryostat fig. B

2.4. Resistance
Scheme fig. C
Sample holder and sample mounting fig. D

2.5. X-Ray diffraction patterns

Reference patterns for $YBa_2Cu_3O_{7-\delta}$ tetra, ortho, and Y_2BaCuO_5 on figs E, F, G



A_s = cross sectional area of the pick-up coil
N_s = number of turns
f = filling factor

$$V_{IND} = -\frac{d\Phi}{dt} \propto -\frac{d}{dt}\left[N_s A_s\left(\mu_0 H + \mu_0 f M\right) - N_s A_s \mu_0 H\right]$$

$$= -\mu_0 N_s f A_s \cdot \frac{dM}{dt} = -\mu_0 N_s f A_s \cdot \frac{dM}{dH}\cdot\frac{dH}{dt}$$

$$= \left\{ H = H_0 e^{i\omega t} \right.$$

$$= -i\mu_0\omega H \cdot N_s f A_s \cdot \frac{dM}{dH}.$$

$$\boxed{V_{IND} \sim \chi}$$

fig B

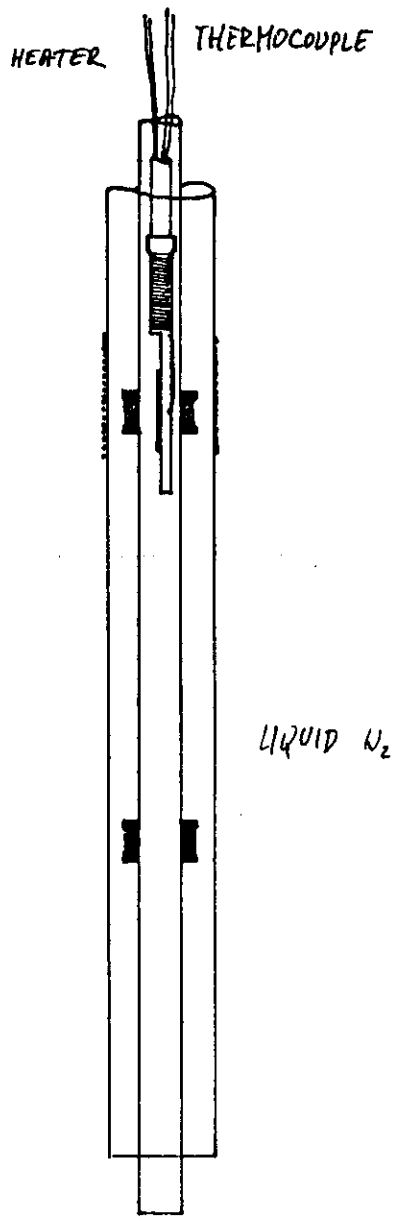


Fig C

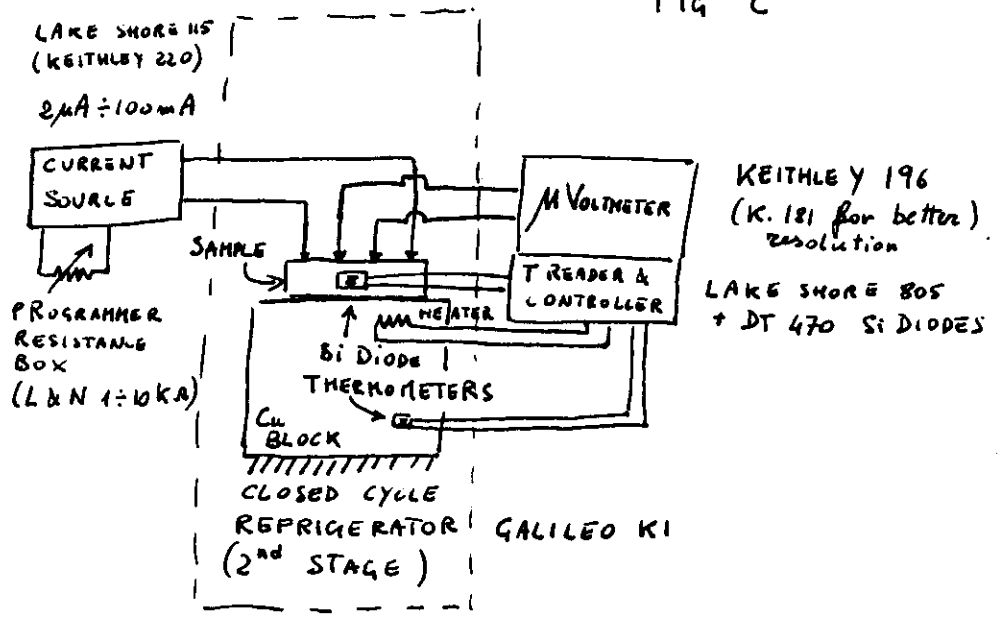
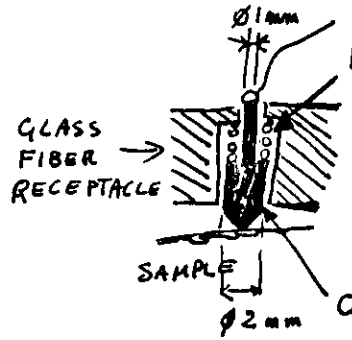


FIG. D



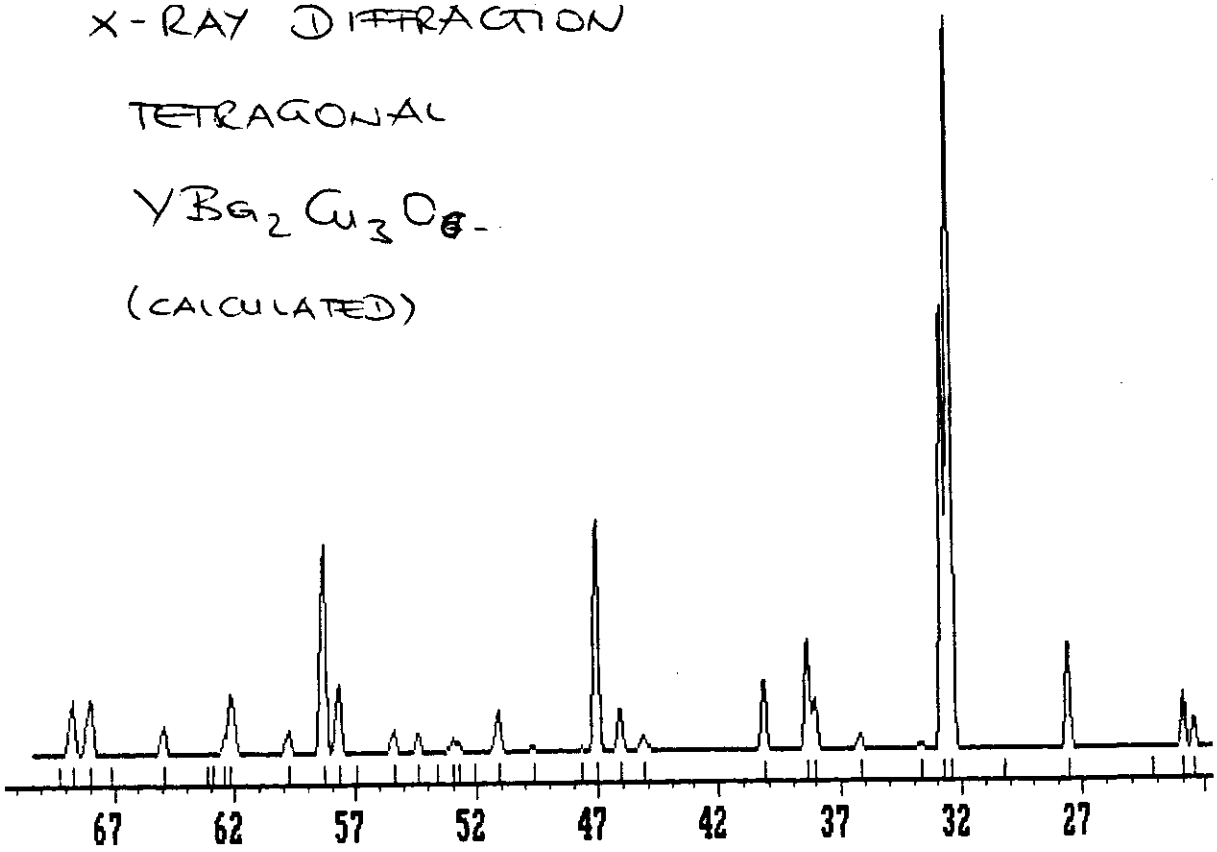
SPRING LOADED
MECHANICAL CONTACTS
(Typ. contact RESISTANCE ~ 1Ω
at any T)

X-RAY DIFFRACTION

TETRAGONAL

$YBa_2Cu_3O_{6-}$

(CALCULATED)

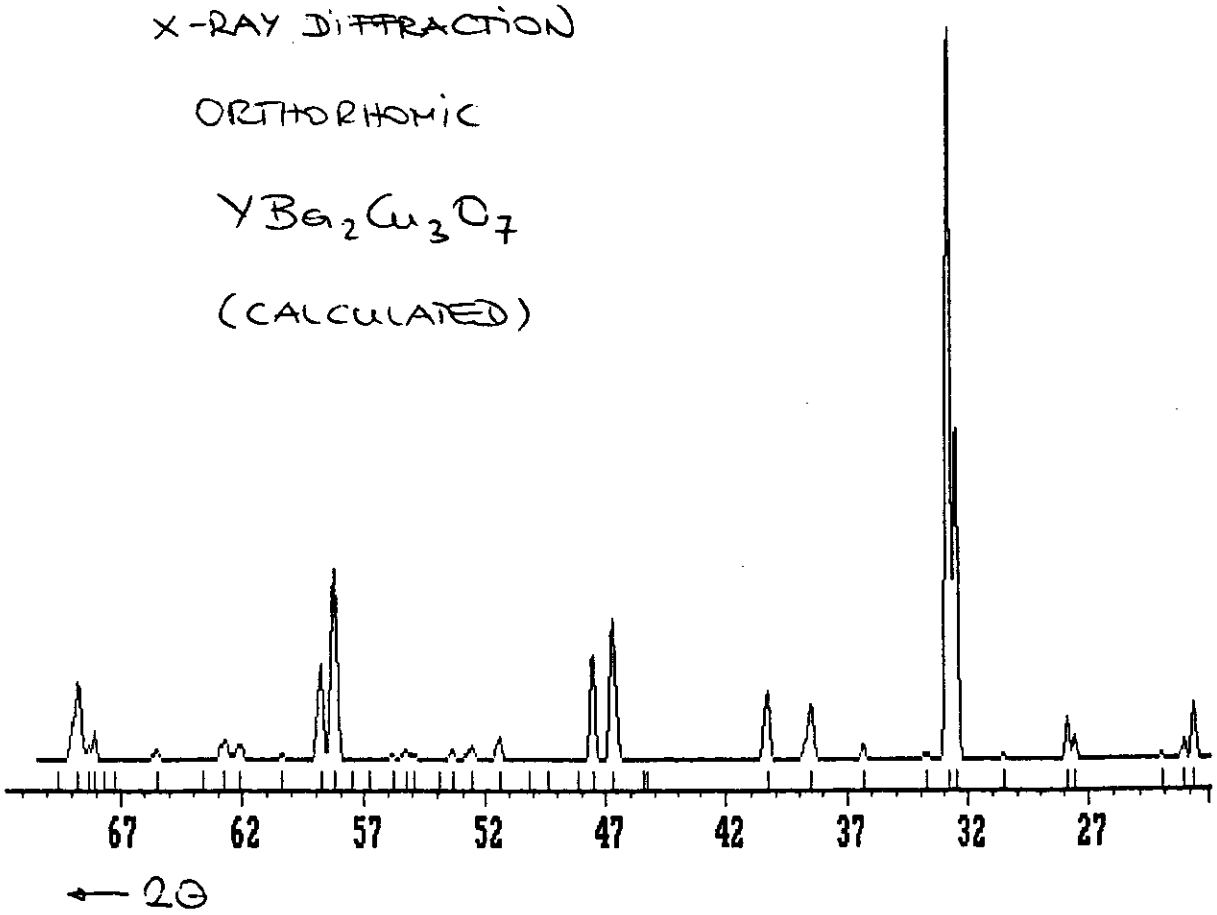


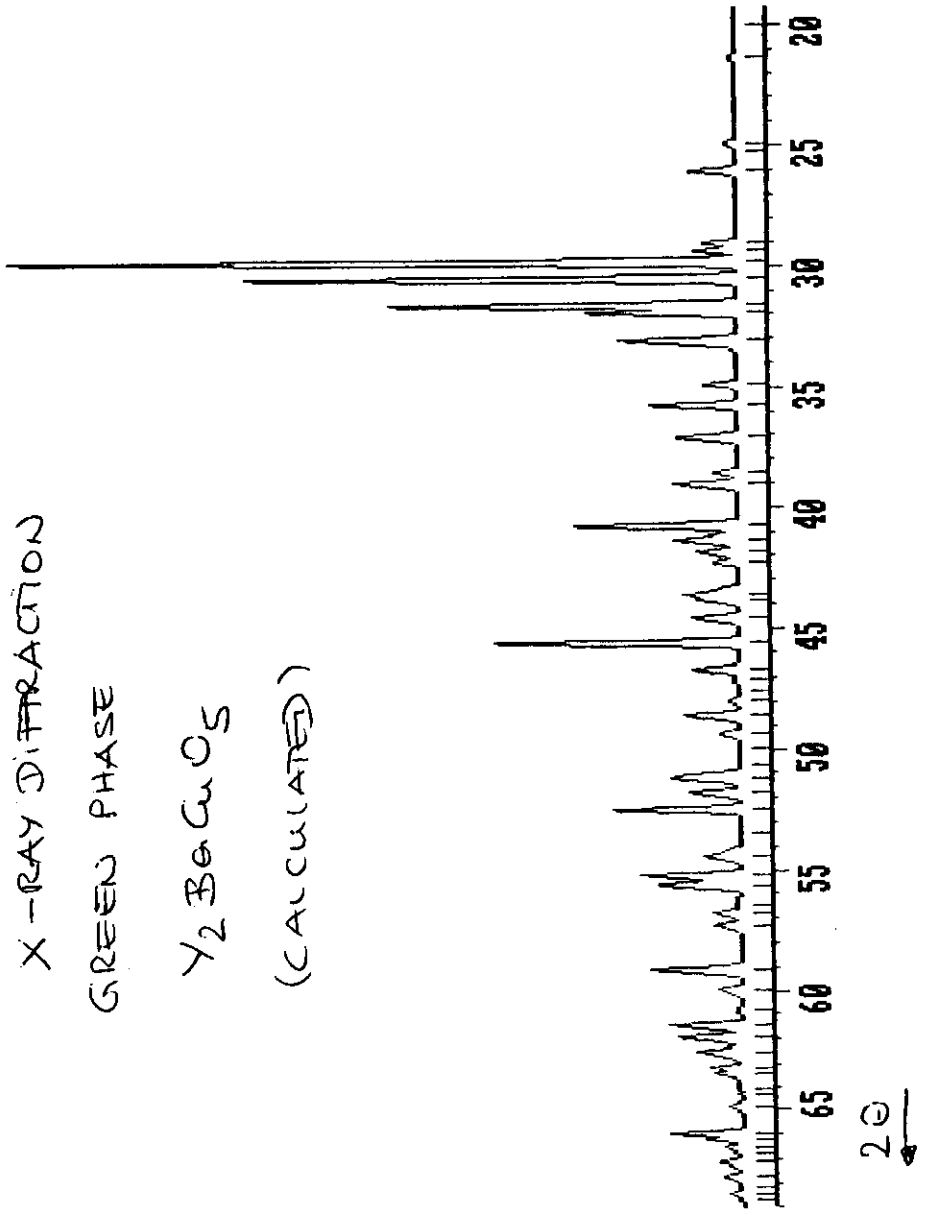
X-RAY DIFFRACTION

ORTHORHOMIC

$YBa_2Cu_3O_7$

(CALCULATED)





X-RAY DIFFRACTION
GREEN PHASE
Y_2BaCuO_5
(CALCULATED)

TABLE I

DENSITY MEASUREMENT
AFTER 3RD HEAT TREATMENT
AT 930°C OF $YBa_2Cu_3O_{7-\delta}$

SAMPLE	GROUP	DENSITY [g/cm³]
$YBa_2Cu_3O_{7-\delta}$	1.5	3.835
"	1.1	4.421
"	1.6	3.833
"	1.4.1	4.30
"	1.4.2	3.965
"	2.1	4.22
"	2.5	4.44
"	2.5	4.44
" (5 tons)	2.4	4.243
" (10 tons)	2.4	4.52
" (10 tons)	2.4	4.80
" (10 tons)	2.4	4.71
" (5 tons)	2.4	4.65

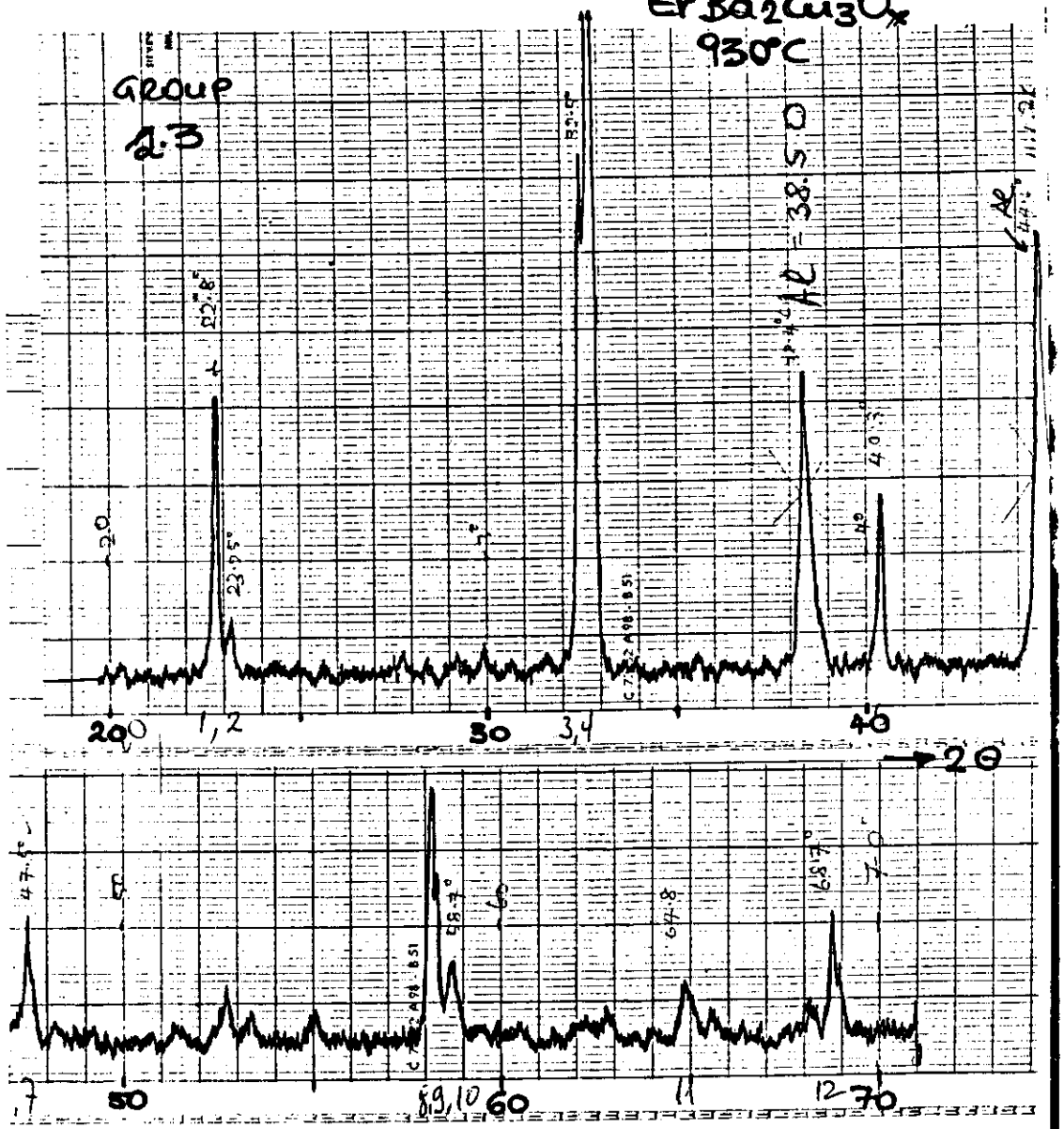
TABLE 2

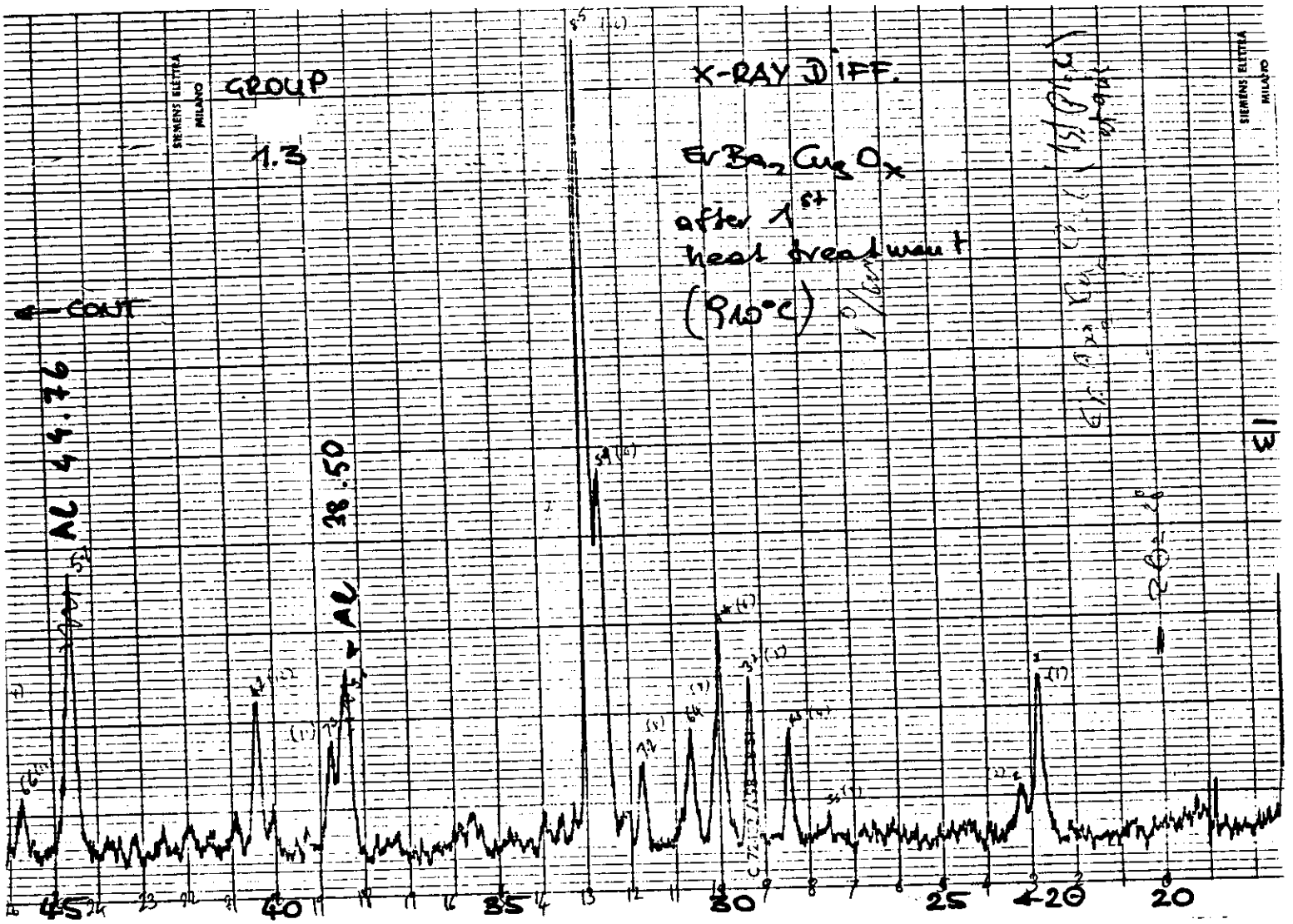
DENSITY MEASUREMENTS
AFTER 3RD HEAT TREATMENT
AT 875°C OF $Bi_2Ca_1Sr_2Cu_2O_x$

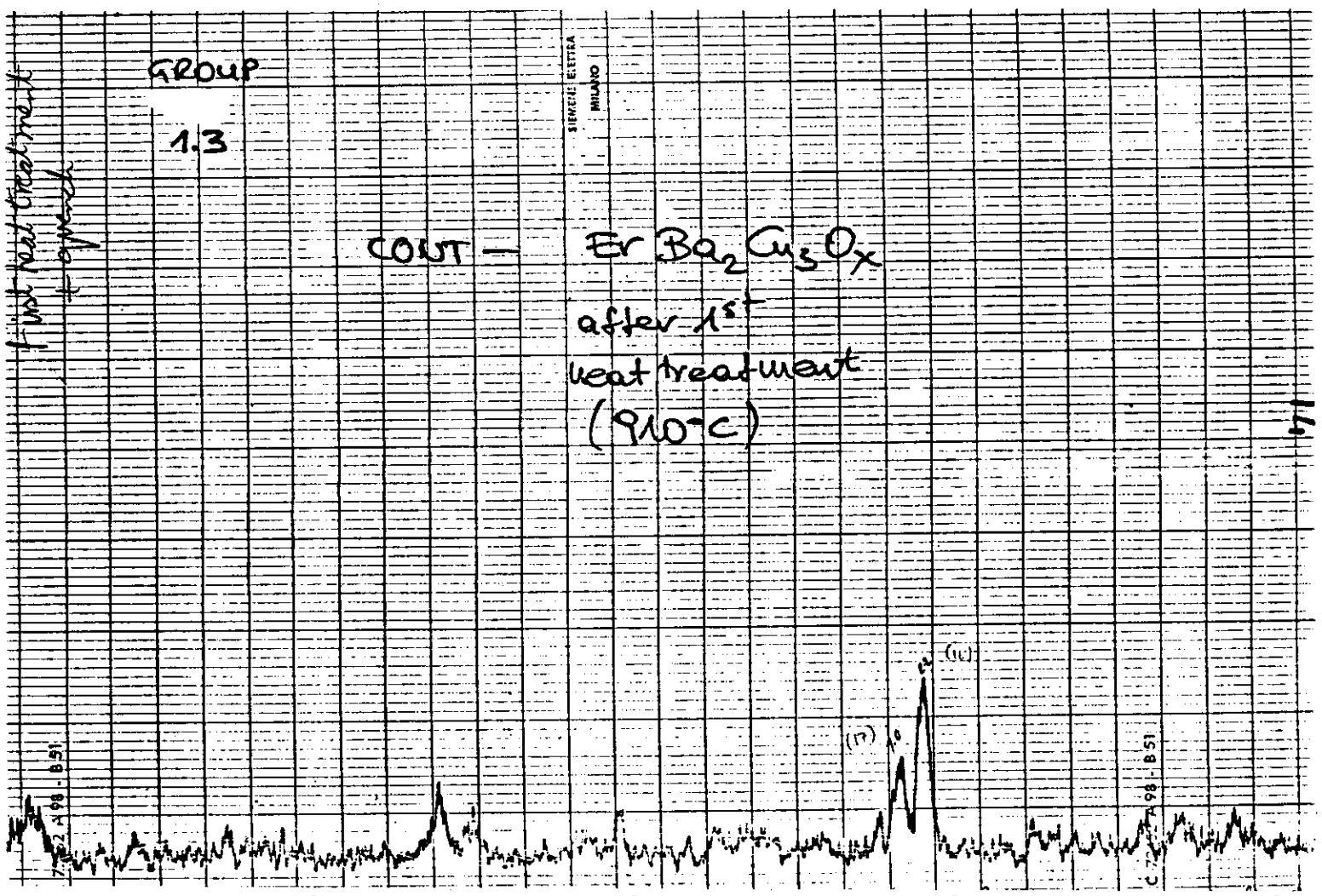
SAMPLE	DENSITY [g/cm³]
1	3.56
2	4.27
3	3.37
4	3.44
5	3.45
6	3.54
7	3.41
8	3.48
9	3.42
10	3.48
11	3.36

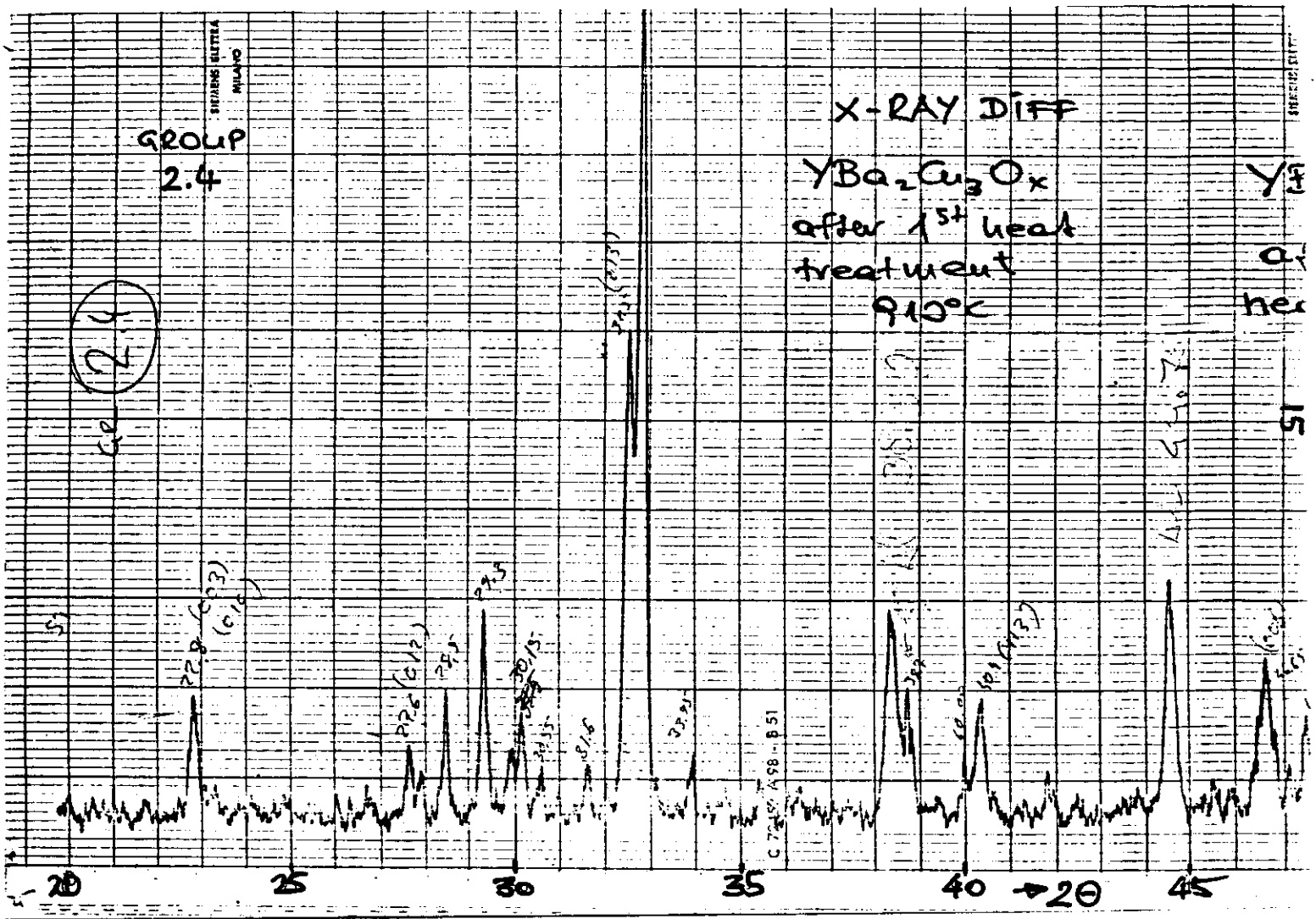
X-RAY DIFFR.

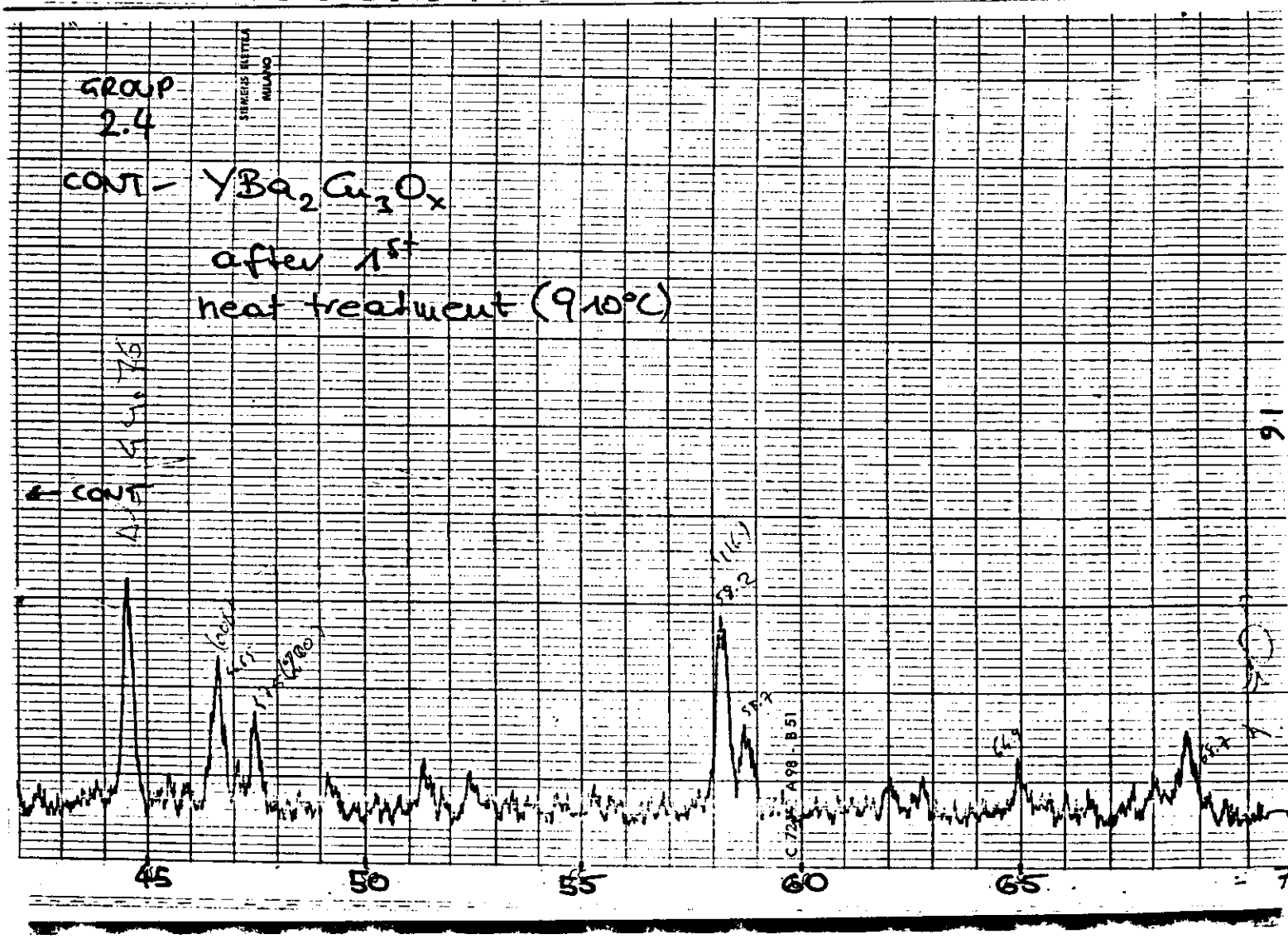
$ErBa_2Cu_3O_x$
930°C

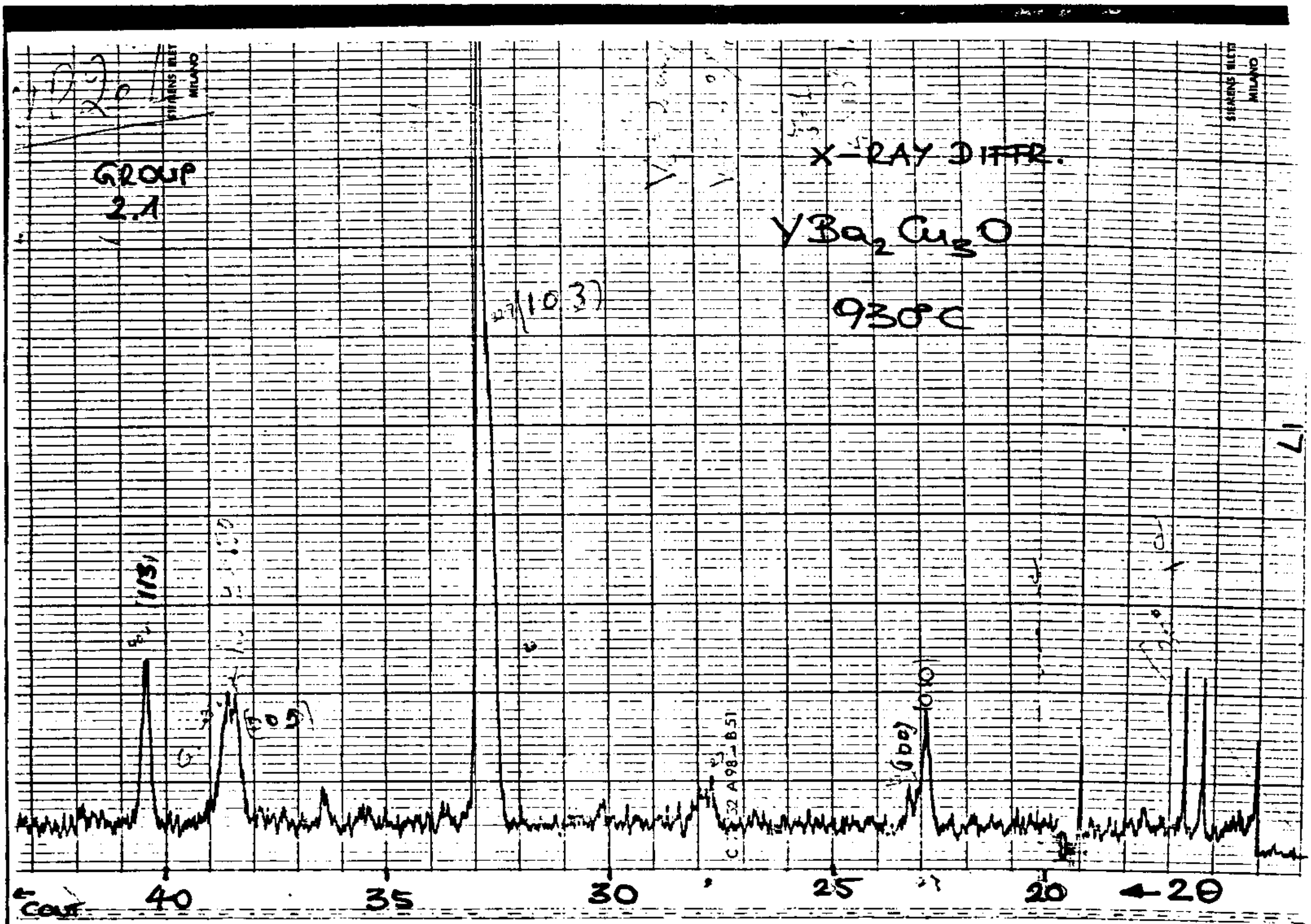


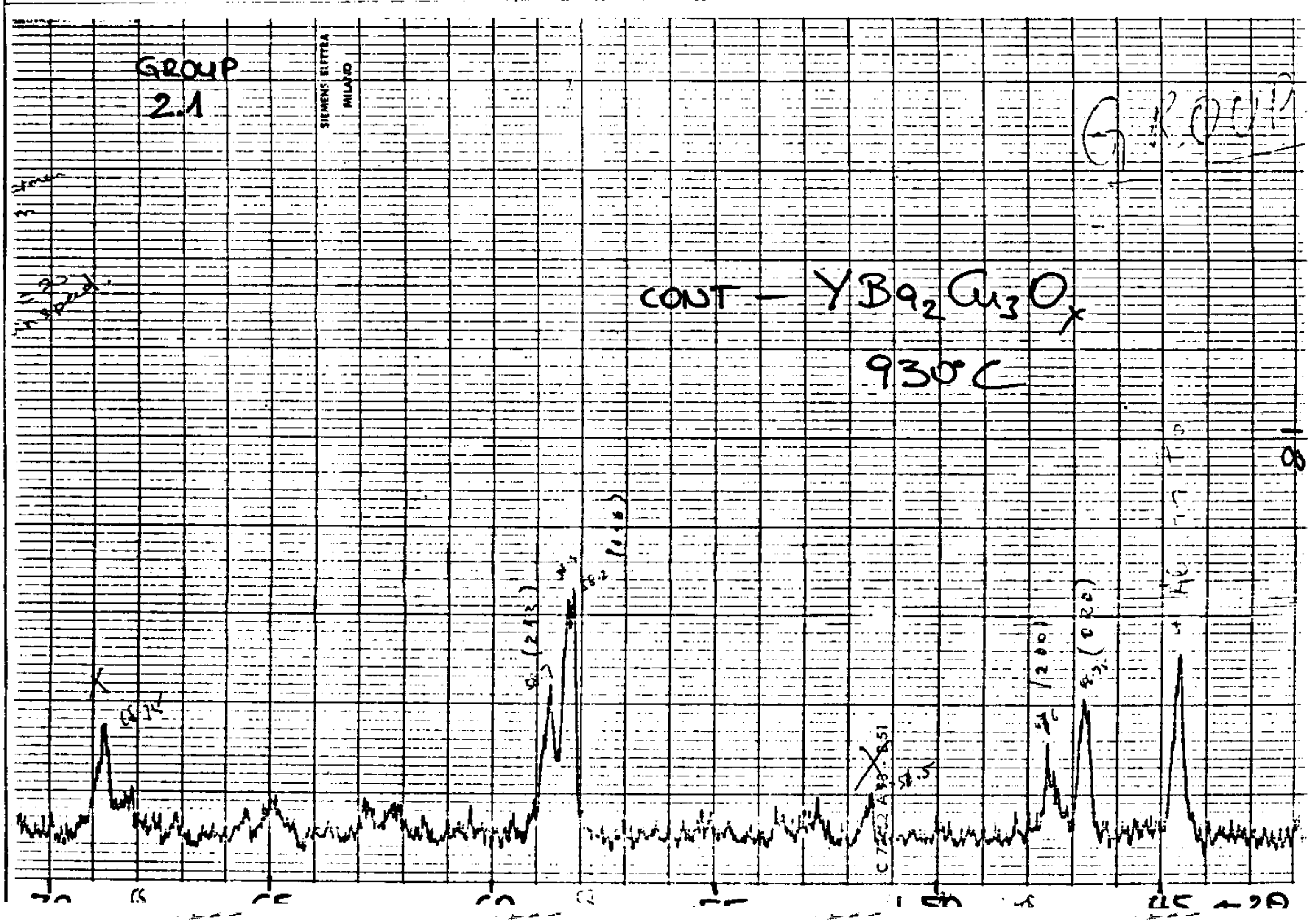

SIEMENS ELETTRA MILANO
GROUP
1.3
X-RAY DIFF.
$ErBa_2Cu_3O_x$
after 1st
heat treatment
(910°C)
COUT
AL 44.76
38.50
AL
45
40
35
30
25
20
2Θ


First heat treatment + anneal
GROUP
1.3
SIEMENS ELETTRA MILANO
COUT
$ErBa_2Cu_3O_x$
after 1st
heat treatment
(910°C)

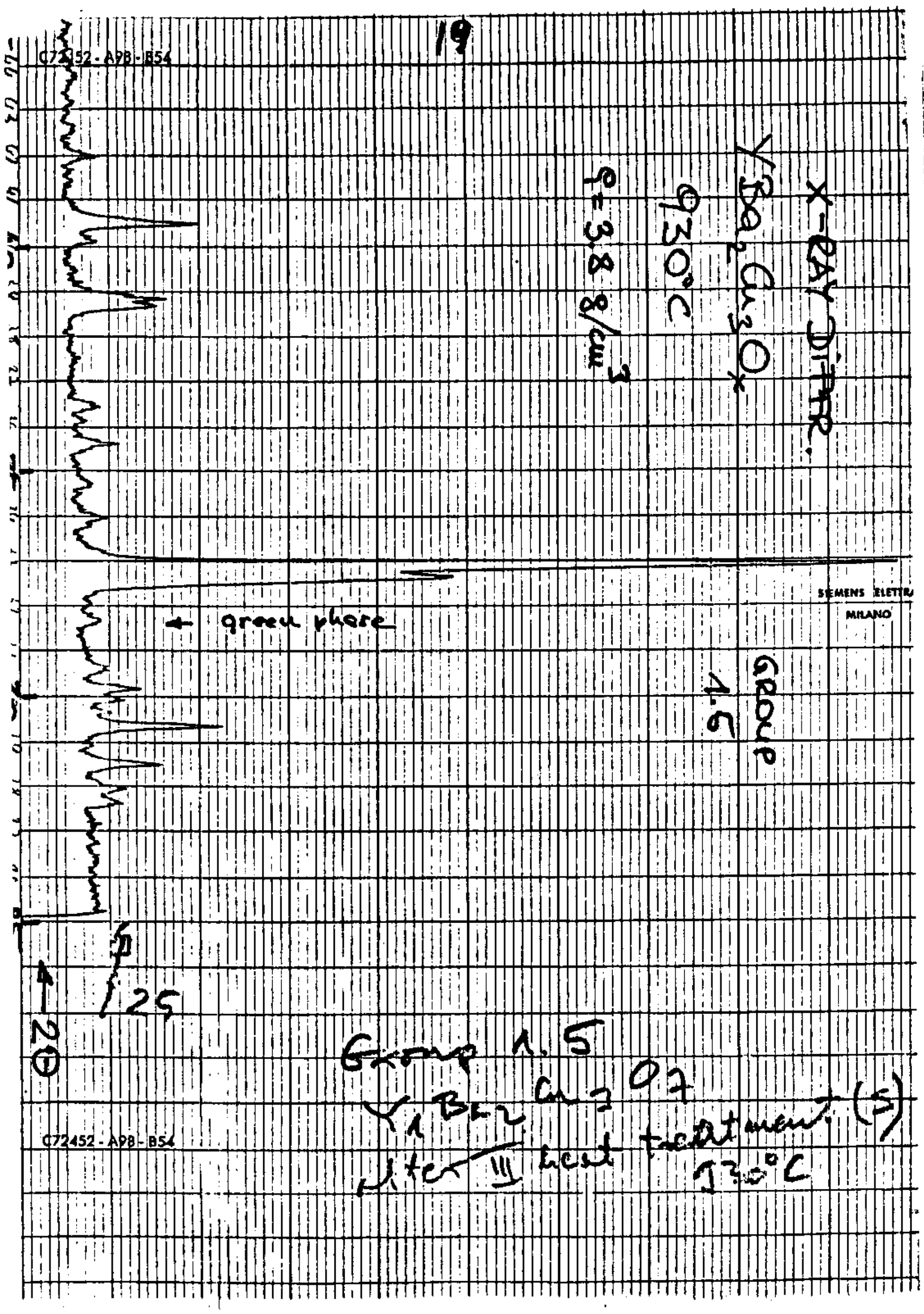

GROUP
2.4
X-RAY DIFF
$YBa_2Cu_3O_x$
after 1st heat
treatment
910°C
SIEMENS ELETTRA MILANO
20
25
30
35
40
45
→2θ


GROUP
2.4
CONT- $YBa_2Cu_3O_x$
after 1st
heat treatment (910°C)
SIEMENS ELETTRA MILANO
← CONT
45
50
55
60
65

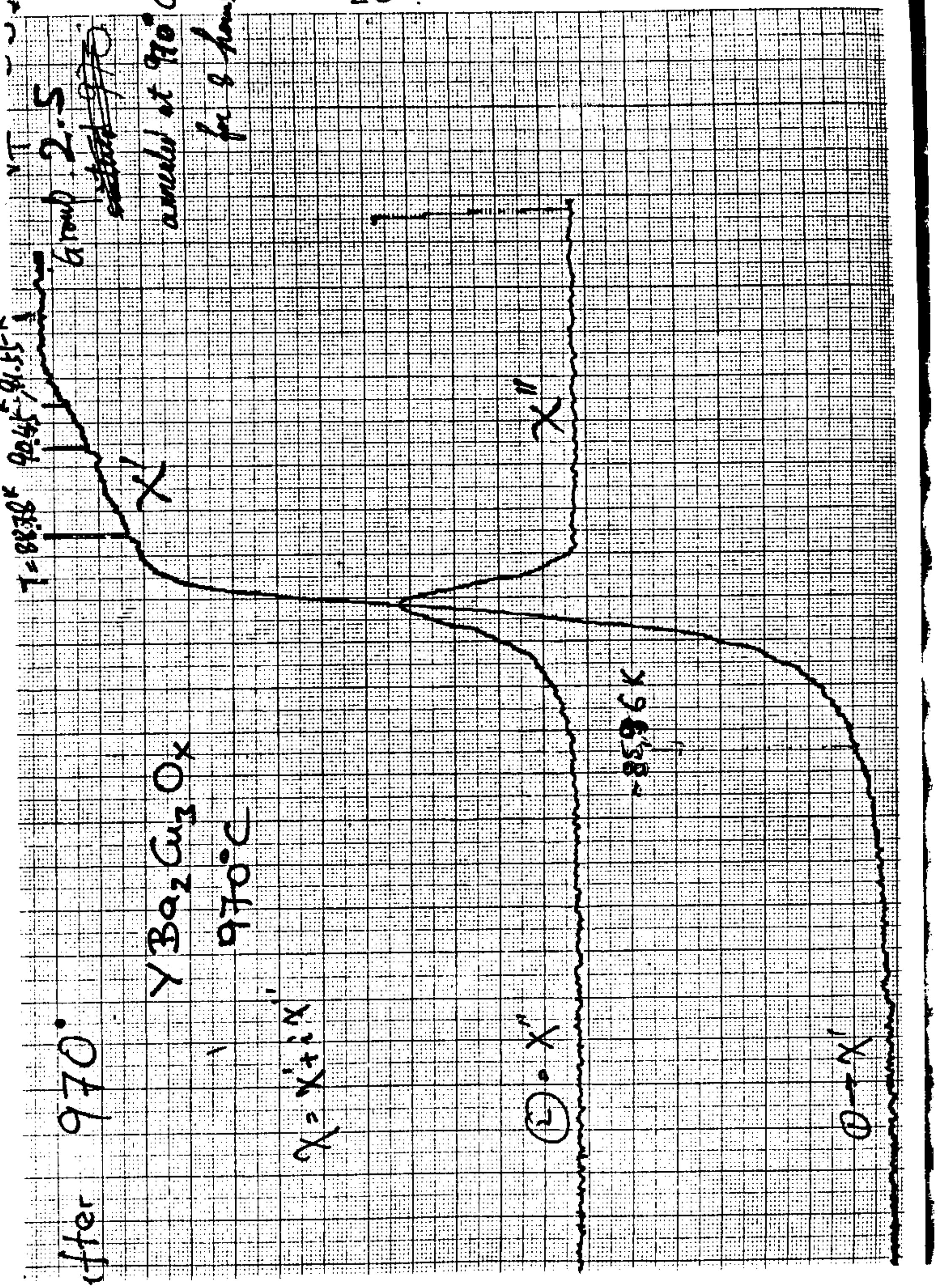

17
X-RAY DIFFR.
YBa_2Cu_3O
930°C
GROUP 2.1
(113)
(103)
(005)
(100)
(010)
40
35
30
25
20
← 2θ
← CONT
SIEMENS ELET MILANO
C 7 52 A 98 - B 51

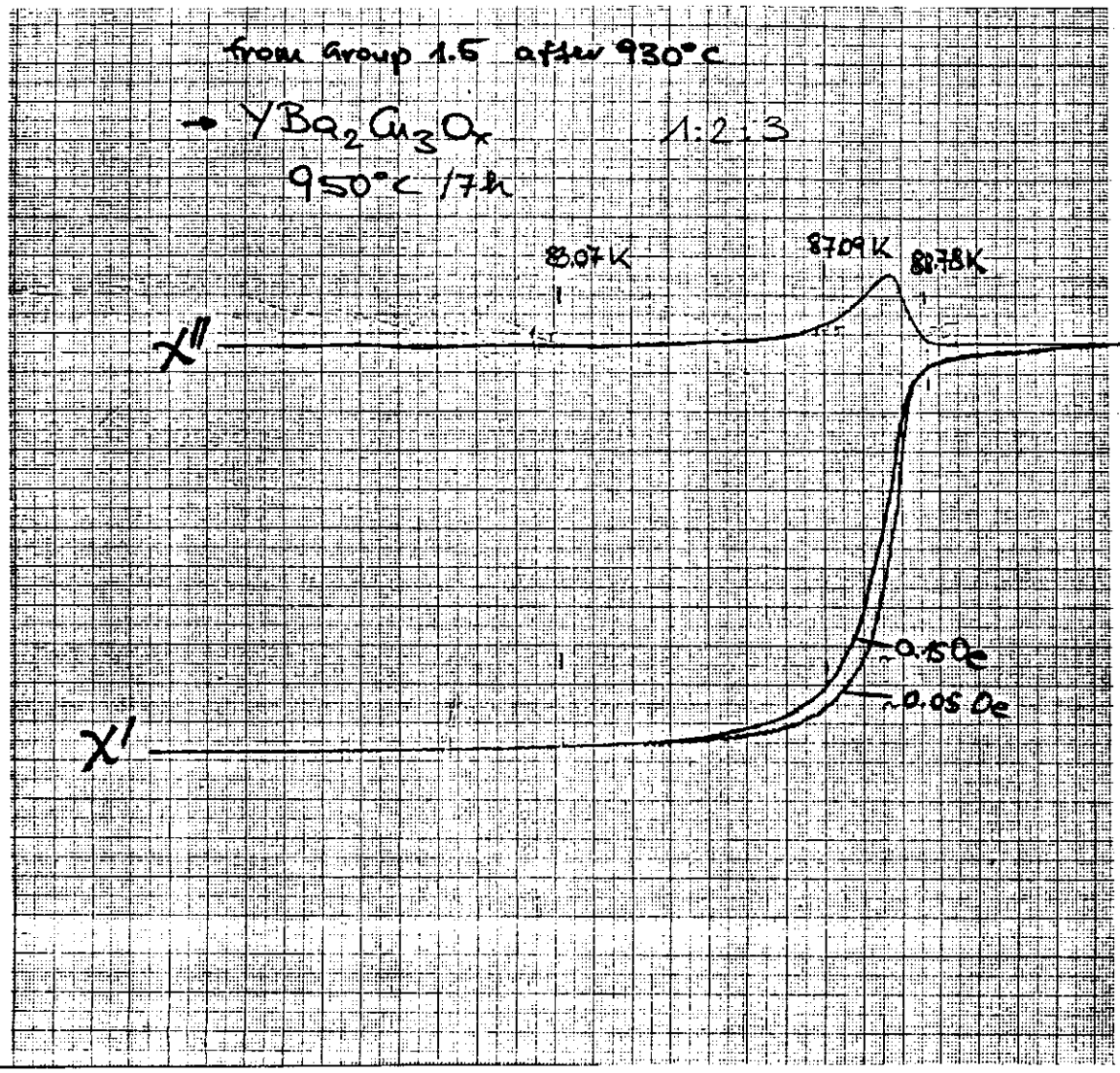

18
GROUP 2.1
CONT — $YBa_2Cu_3O_x$
930°C
(212)
(116)
(200)
(020)
SIEMENS ELETTRA MILANO
C 7 52 A 98 - B 51

19

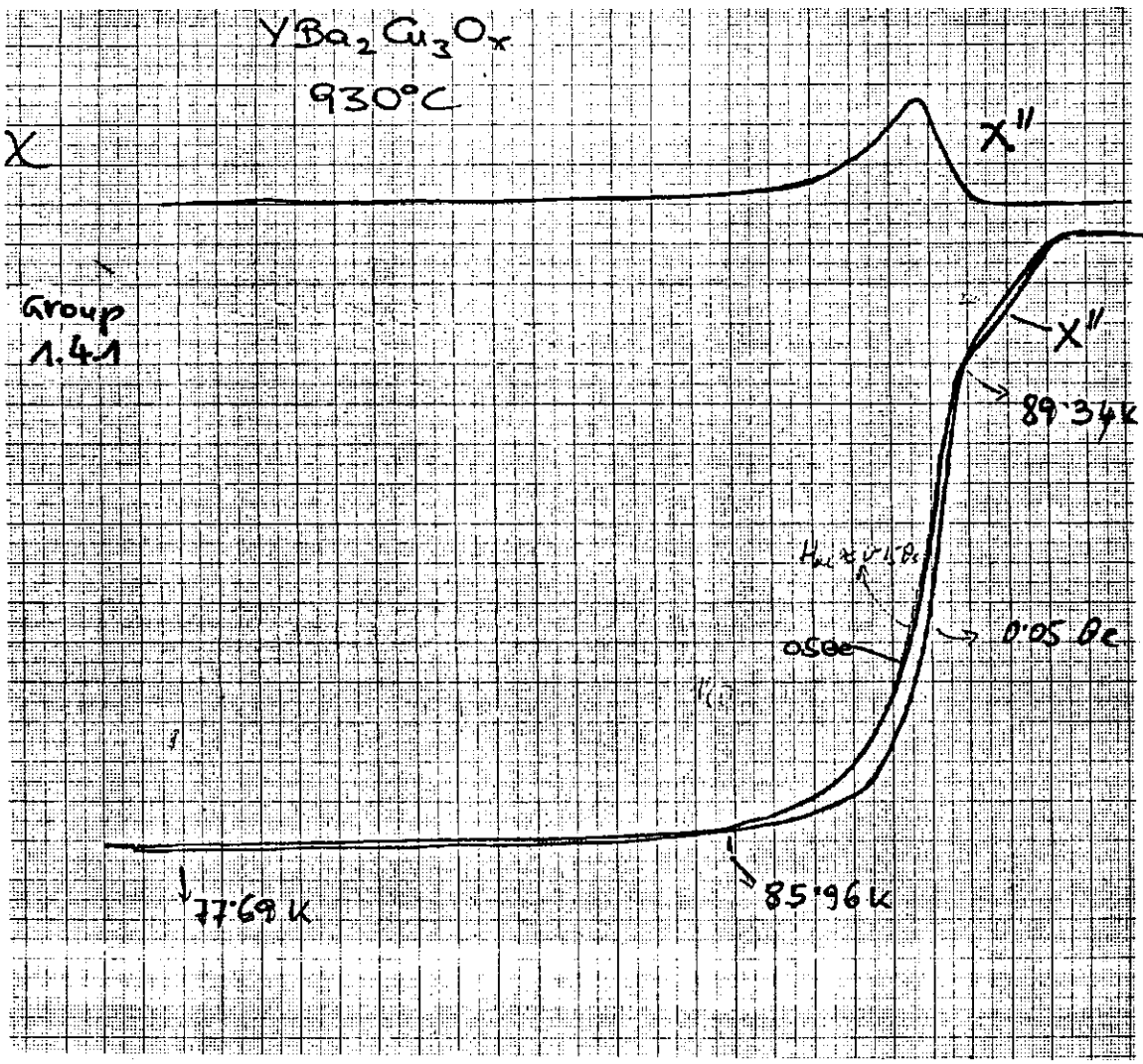

C72452 - A98 - B54
X-RAY DIFFR.
$YBa_2Cu_3O_x$
930°C
ρ = 3.8 g/cm³
GROUP
1.5
SIEMENS ELETTRA
MILANO
← green phase
25
← 2Θ
Group 1.5
$Y_1 Ba_2 Cu_3 O_7$
after III heat treatment (S)
930°C
C72452 - A98 - B54

20 !

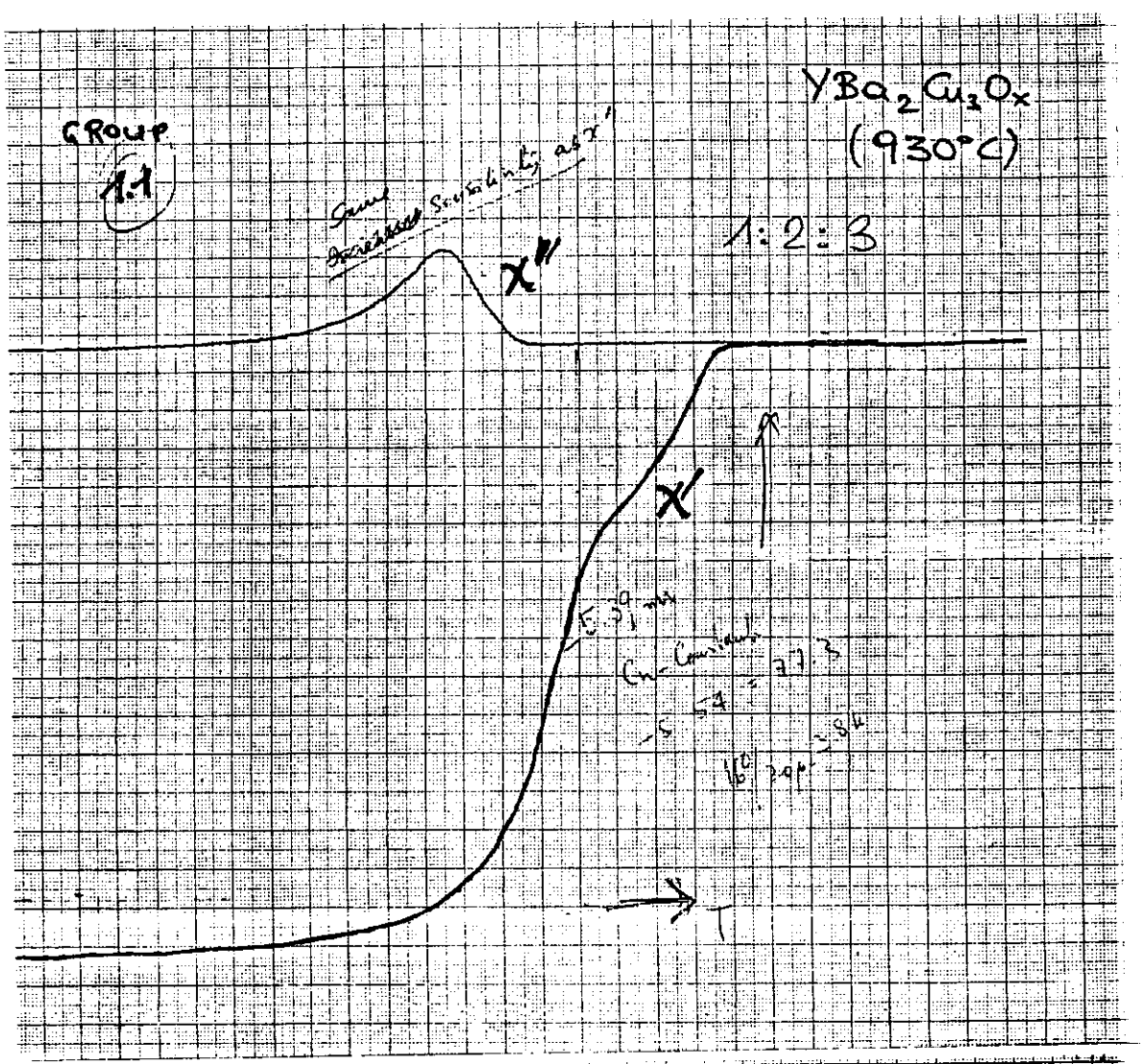

Group 2.5
annealed at 970°C
for 8 hours
X''
X'
T = 88.38 K
90.45 K
91.55 K
88.38 K
$YBa_2Cu_3O_x$
970°C
$\chi = \chi' + i\chi''$
after 970°


from Group 1.5 after 930°C
→ $YBa_2Cu_3O_x$ 1:2:3
950°C / 7h
88.07K
87.09K
88.78K
χ''
~0.5 Oe
~0.05 Oe
χ'

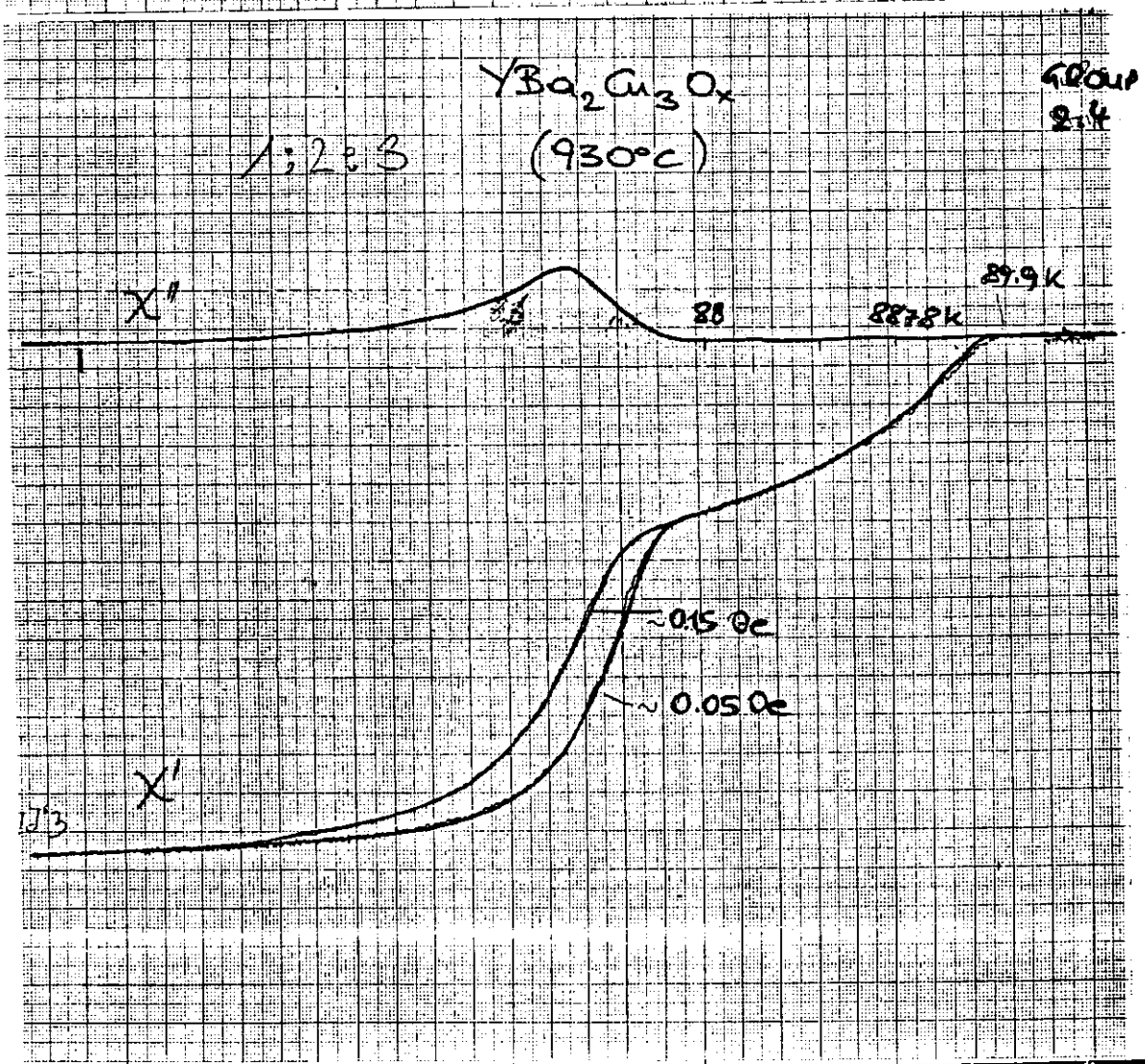
21 !


$YBa_2Cu_3O_x$
930°C
χ
χ''
Group
1.4.1
χ''
89.34K
0.5 Oe
0.05 Oe
77.69 K
85.96 K

22 *

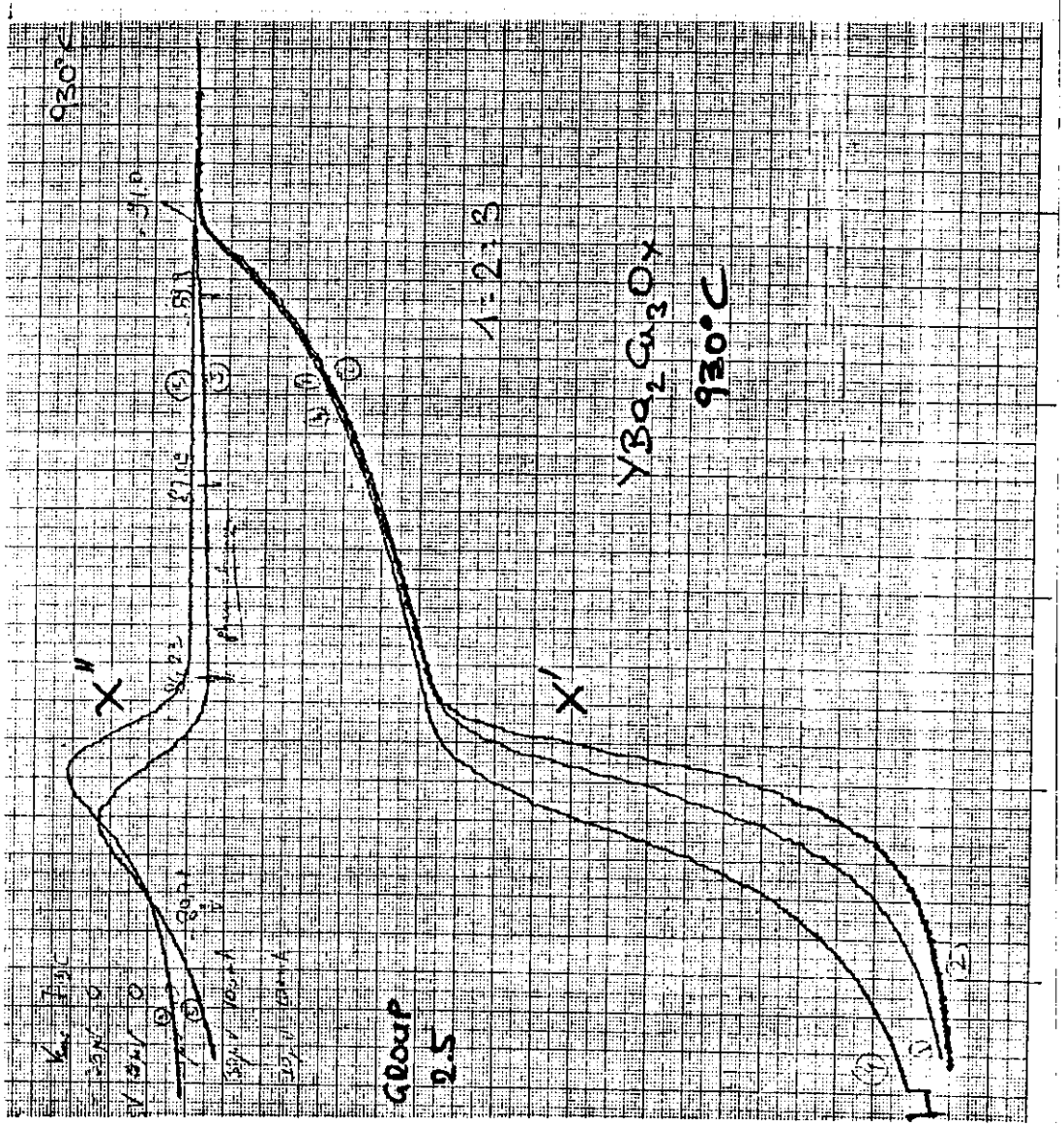

GROUP
1.1
$YBa_2Cu_3O_x$
(930°C)
1:2:3
X''
X'

23 *

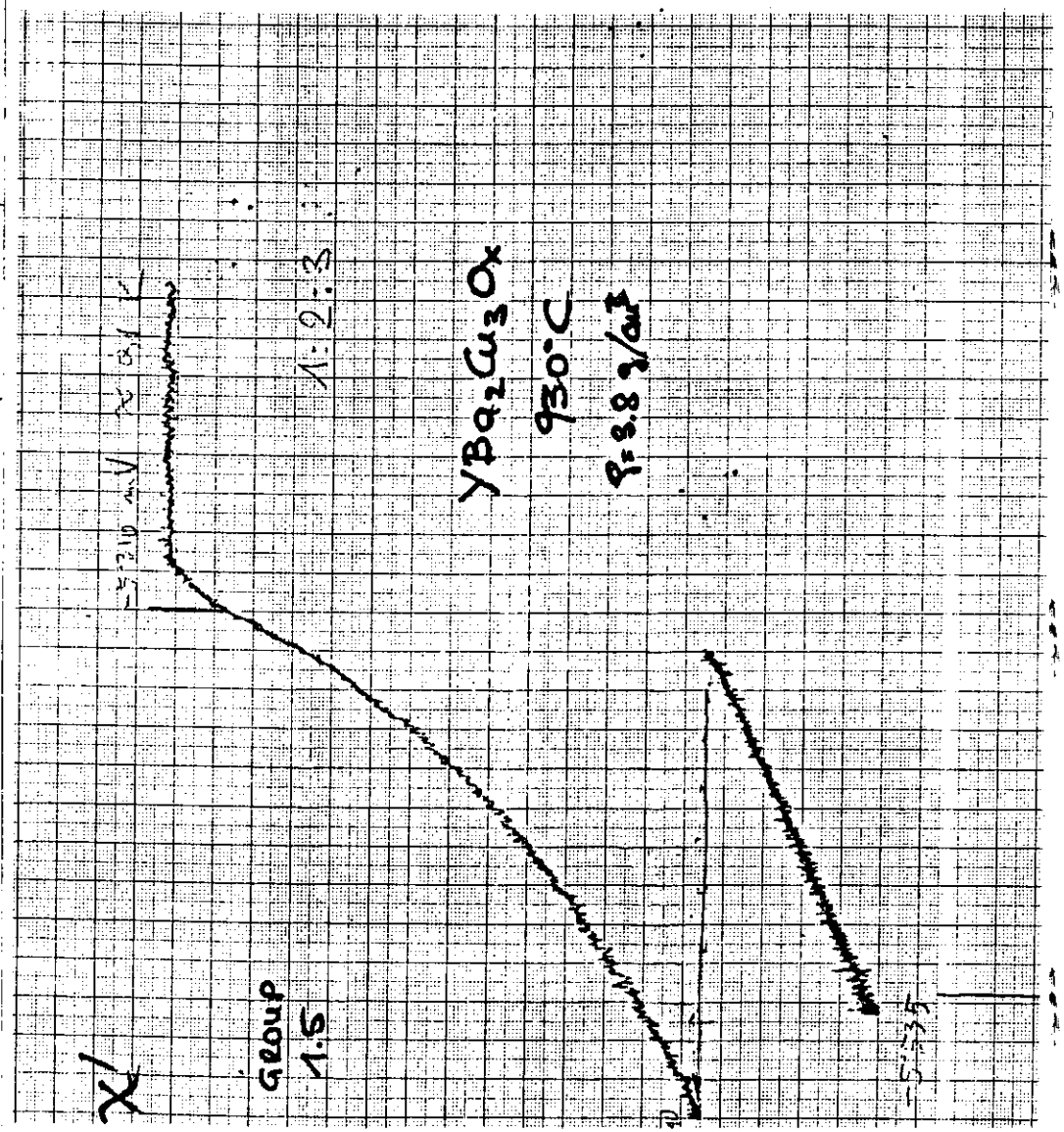

$YBa_2Cu_3O_x$
(930°C)
1:2:3
GROUP
2.4
X''
89.9K
88
88.8K
~0.15 Oe
~0.05 Oe
X'

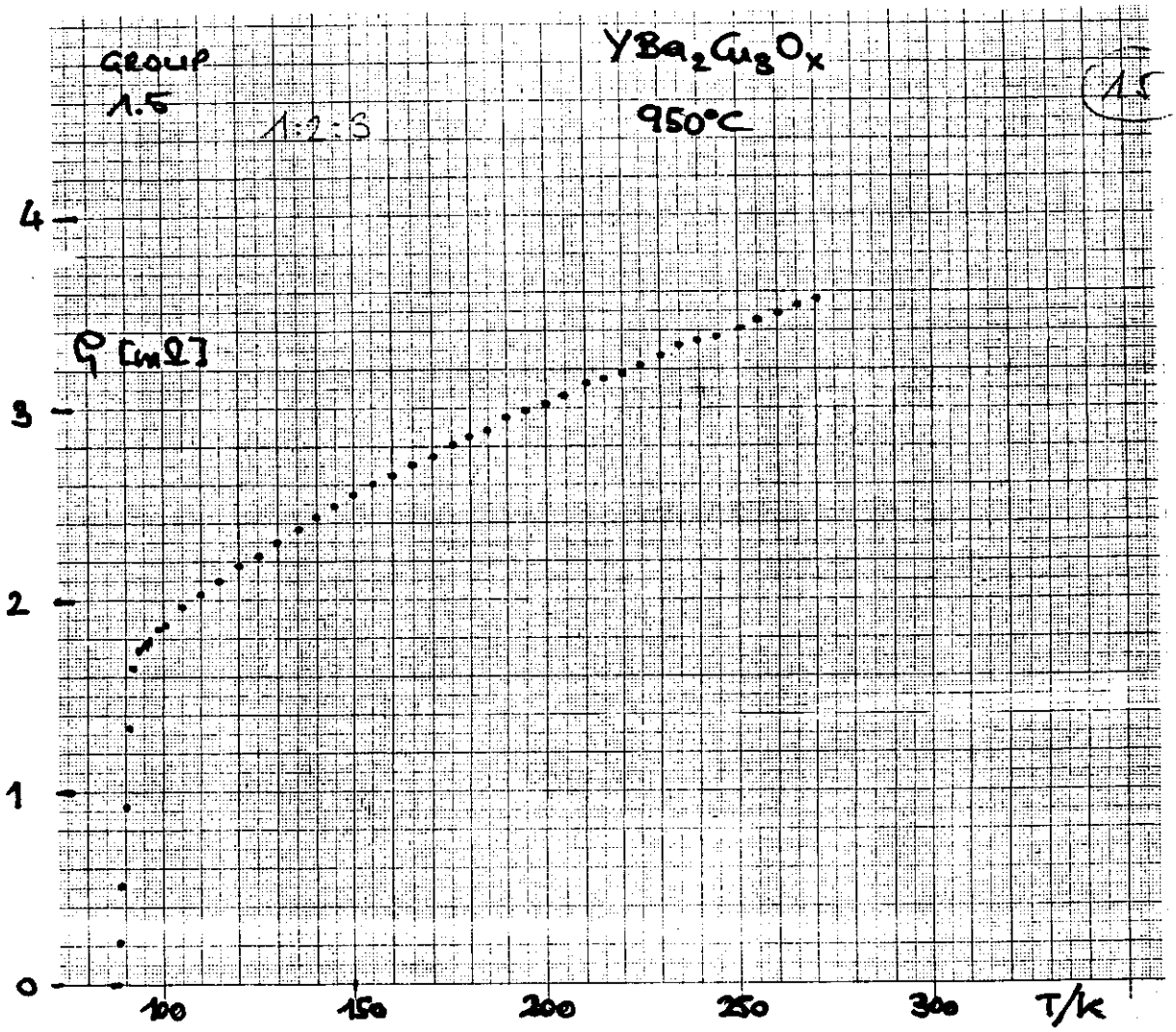
24 *

25 *

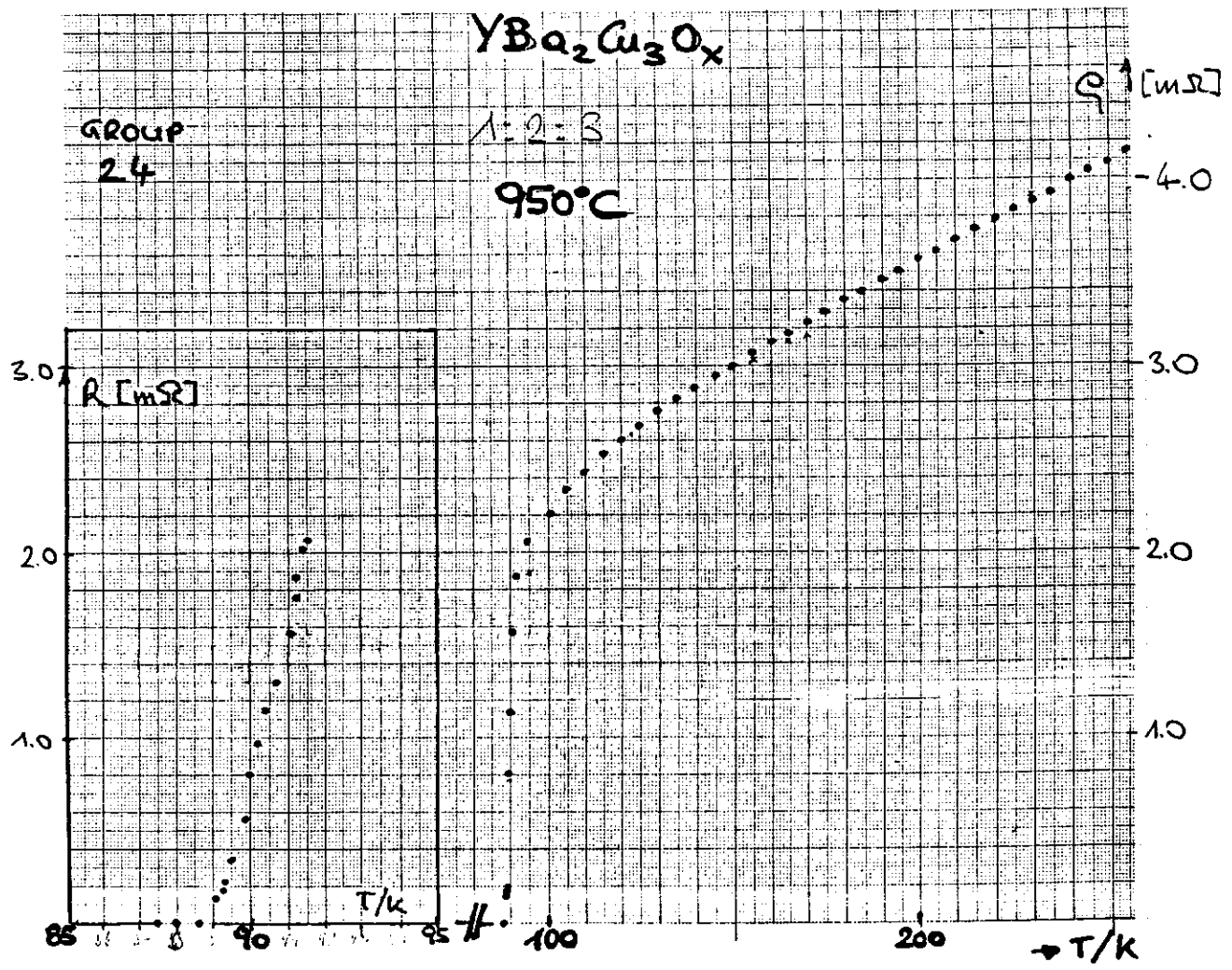

930°C
X''
X'
YBa2Cu3Ox
930°C
1:2:3
GROUP
2.5

26 *

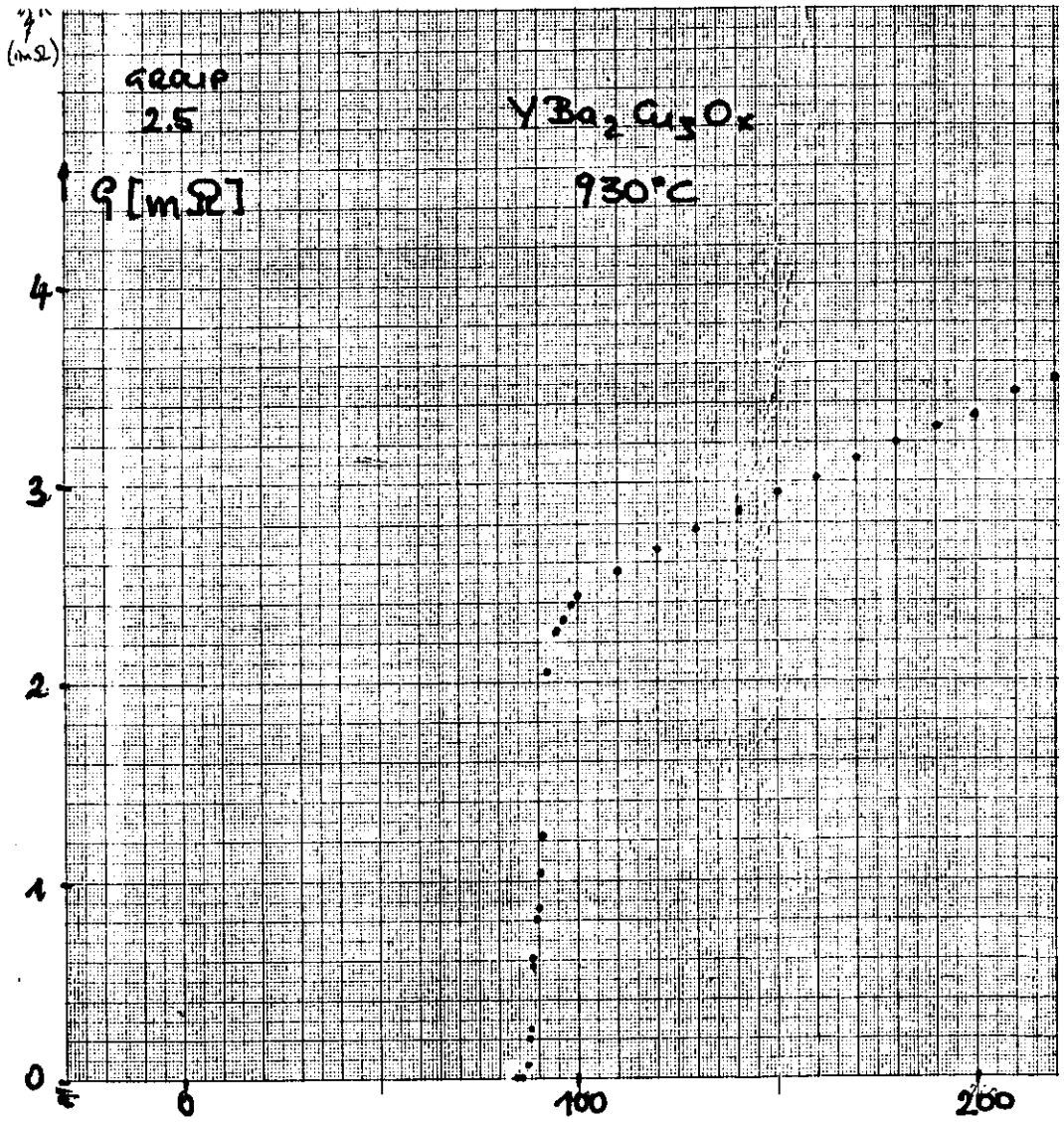

X'
GROUP
1.5
YBa2Cu3Ox
930°C
1:2:3

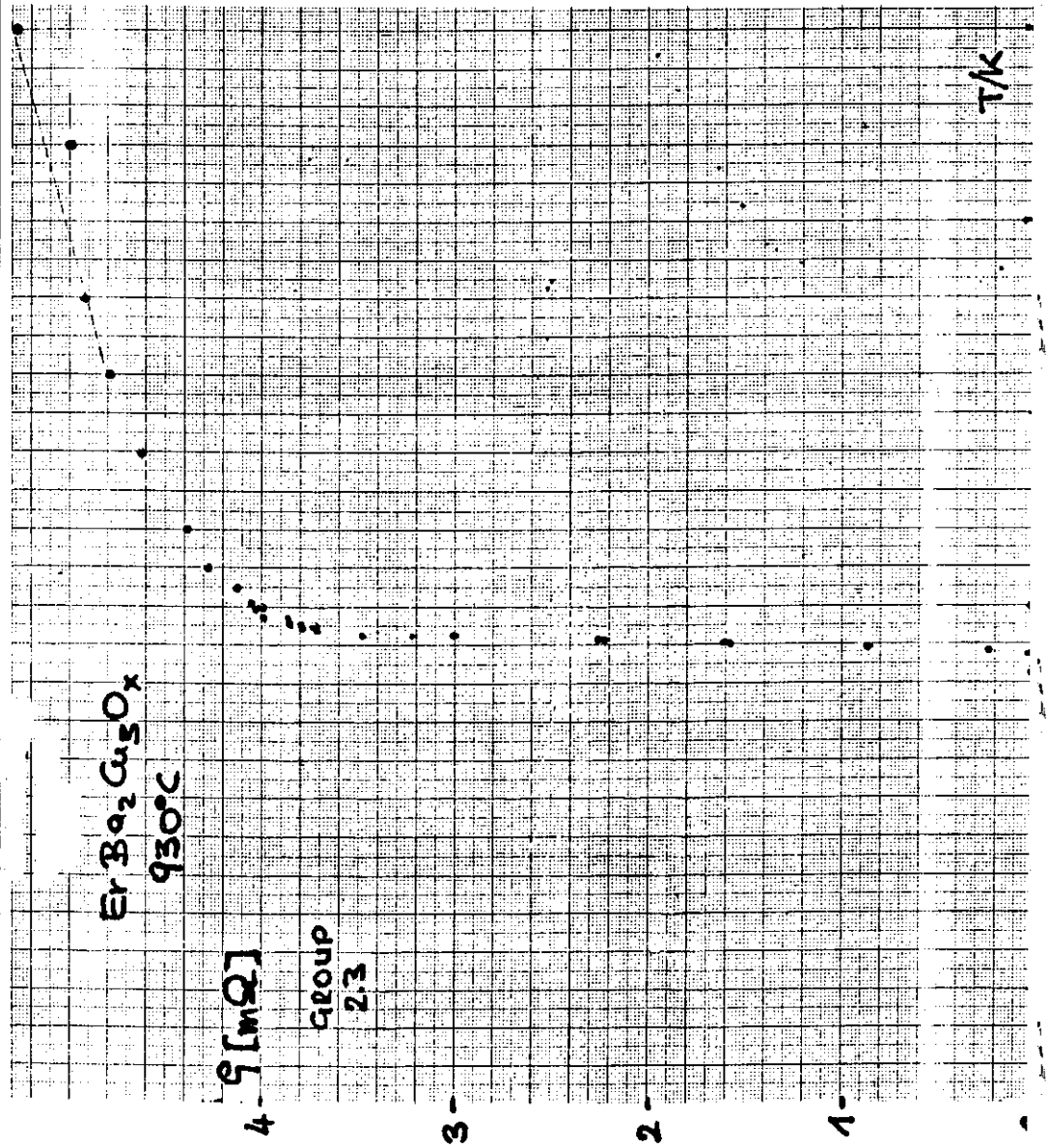

GROUP
1.5
1:2:3
$YBa_2Cu_3O_x$
950°C
1.5
ρ [mΩ]
4
3
2
1
0
100
150
200
250
300
T/K

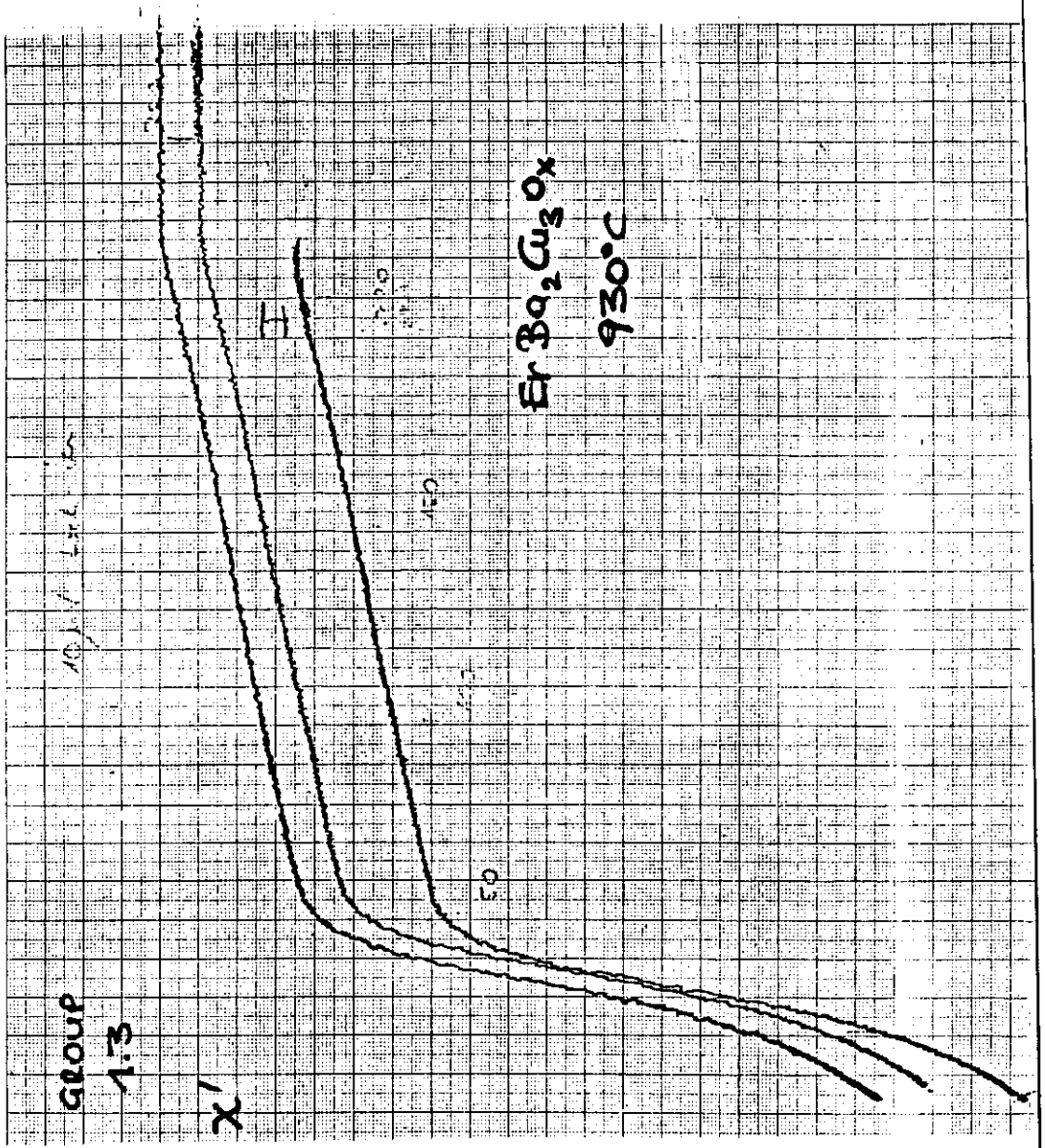
27 !

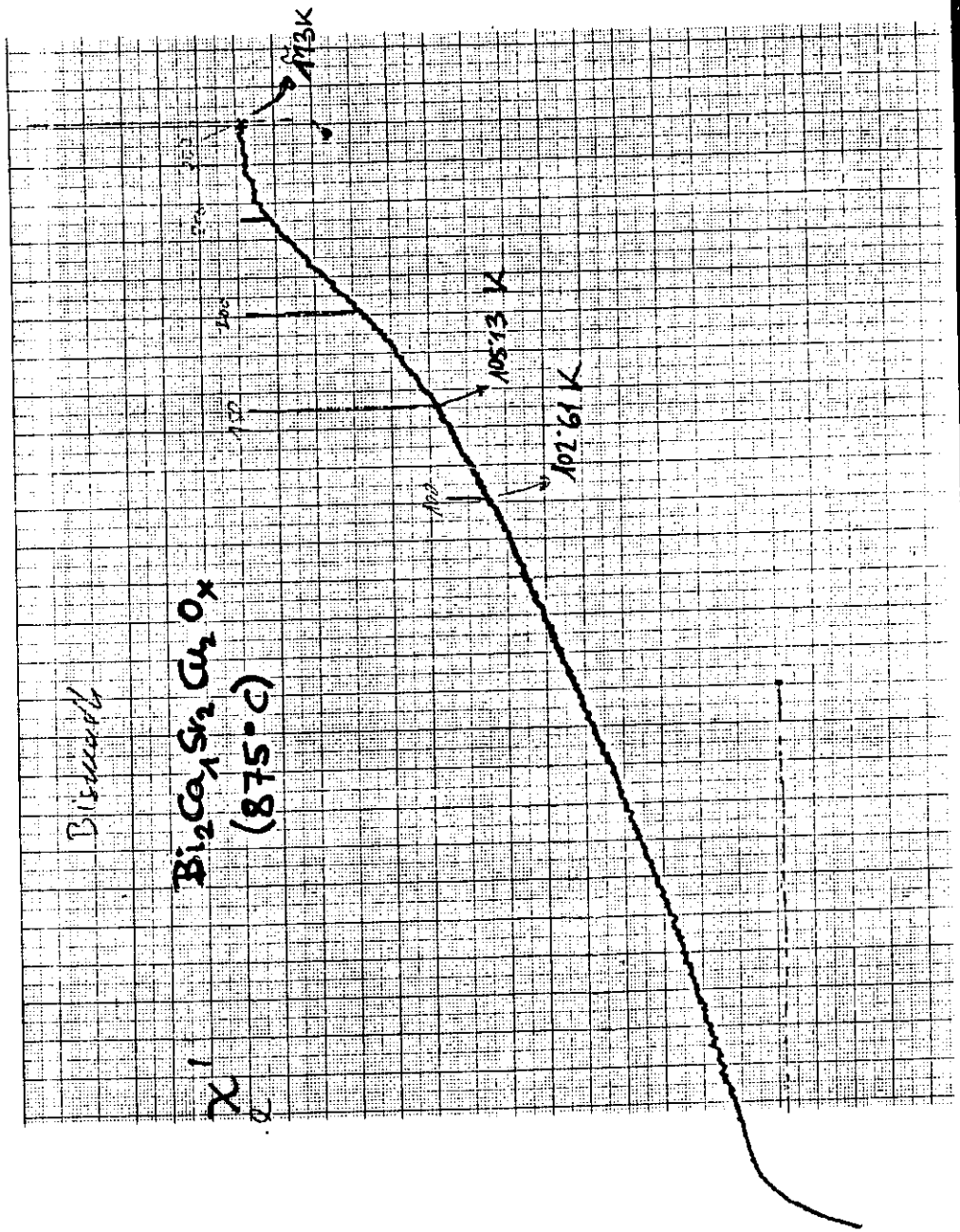

$YBa_2Cu_3O_x$
GROUP
24
1:2:3
950°C
ρ [mΩ]
4.0
3.0
2.0
1.0
R [mΩ]
3.0
2.0
1.0
T/K
85
90
95
100
200
T/K

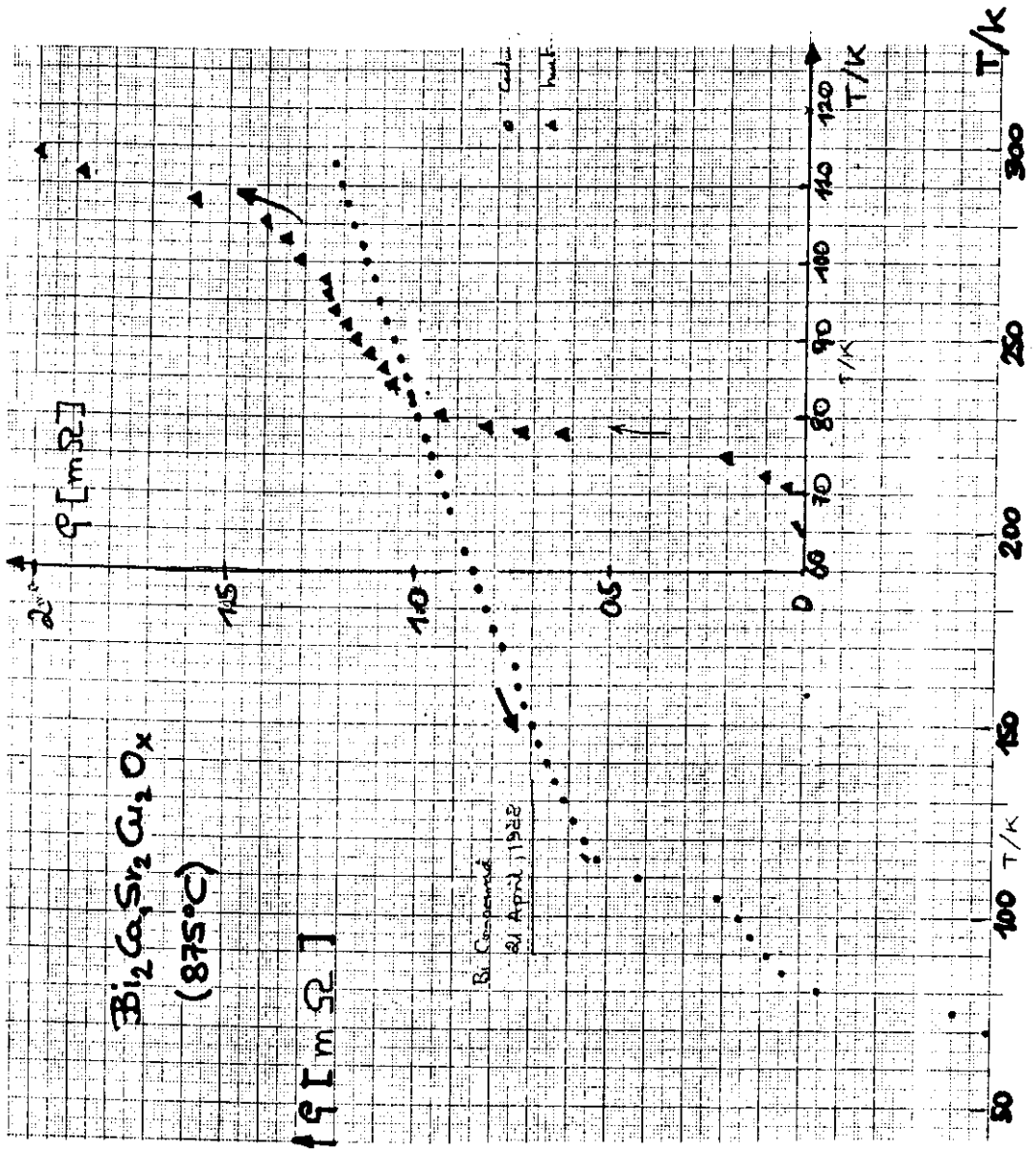
28 !


ρ
(mΩ)
group
2.5
YBa2Cu3Ox
930°C
ρ [mΩ]
4
3
2
1
0
0
100
200


T/K
ErBa2Cu3Ox
930°C
group
2.3
ρ [mΩ]
4
3
2
1


GROUP
1.13
χ'
T
Er Ba2 Cu3 Ox
930°C


χ'
T
Bi2 Ca1 Sr2 Cu2 Ox
(875°C)
113K
105·13 K
102·61 K



Officine Galileo
Via Einstein, 35
Campi Bisenzio (FI)
Italy
cryogenerators & vacuum

Keithley Instruments
28775 Aurora Rd
Cleveland, Ohio 44139
Current sources; nanovoltmeters

ARBORE CATALDO & C. Snc
Via Palamanova 127
20132 Milano
quartz furnaces; glass works

BRACCO INDUSTRIA CHIMICA Spa
Via Egidio Folli, 50
20100 Milano
Merck chemicals

BOTTIGELLI & C.Spa
Via Boni, 32
20144 Milano
Hoods; chemical ware

DEGUSSA ITALIA Spa
Via Pisacane 7/B
20016 Pero (MI)
crucibles

EUROTHERM Spa
Viale XXIV Maggio
22070 Guanzate (Como)
Furnaces controls

F.K.V. Srl
Via Baioni, 31/d
24100 Bergamo
Milling equipment

GIBERTINI ELETTRONICA Srl
Via Bellini, 29/31
20096 Novate (MI)
Balances

HELLMA ITALIA Srl
Via A. da Saluzzo, 20/7
20162 Milano
Dies, presses

M.R.C.
Centro Direzionale Colleoni
Pal. Andromeda, Ingresso I
Via Paracelso, 16
20041 Agrate (MI)
Chemicals

INALCO SPA
Via Calabiana, 18
20139 Milano
Janssen Chemicals

EG&G P.AR. INSTR.
P.O. Box 2565
Princeton, NJ 08543 USA
lock-in ampl.

METALLI PREZIOSI Spa
Via Roma 179
20037 Padmo Dugano (MI)
J & M chemicals

W. PABISCH Spa
Via Fratelli Bandiera, 13
20116 Pero (MI)
microscopes

PETRA GIUSEPPE
Strumenti di misura e regolazione
10129 Torino
thermocouples

RIVOIRA
Viale dell'Industria, 36
37135 Verona
L.N_2 cryostats; liquefied gases

SERAFINI
Via Mauro Macchi, 42
20124 Milano
SSL furnaces

LAKESHORE
64E, Walnut Street
Westerville, Ohio 43081 USA
Thermometers, Cryogenics

GRUPPO FLOW
Via Lambro 23
20090 Opera (MI)
Hoods