Erba Instruments Inc.

February 19, 1985

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030-22113
03123

U.S. Nuclear Regulatory
Commission, Region 1
Nuclear Material Section B
631 Park Avenue
King of Prussia, PA 19406

attn: Mr. Lester Tripp

Dear Mr. Tripp:

Please find enclosed the completed form NRC 313 together with the application and a cheque for $210.00 to cover cost for 3L License.

Do not hesitate to contact me if you require any clarification or further information.

Very truly yours,

ERBA INSTRUMENTS, INC.

Tony Bashall

Anthony D. Bashall
Technical Support Manager

ADB/lmm
encls: cheque # 631
application
NRC form 313

cc: TMJackson
file

0315C

"OFFICIAL RECORD COPY"

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REG1 LIC30
29-23484-02 PDR

ML10 FEB 22 1985

APPLICATION FOR REGISTRATION OF
DEVICE CONTAINING RADIOACTIVE SOURCE

ELECTRON CAPTURE DETECTOR

ECD-40

ERBA INSTRUMENTS, INC.

A. SUMMARY PAGE

1. Date of submission: February 15, 1985

2. Device Type: Electron Capture Detector

3. Model: ECD-40

4. Applicant (Distributor): Erba Instruments, Inc.
4 Doulton Place
Peabody, Massachusetts 01960

(Contact): Anthony D. Bashall
Technical Support Manager
(617) 535-5986

5. Other Companies Involved: Carlo Erba Strumentazione S.p.A.
Strada Rivoltana
20090 Rodano (Milan)
PO Box - 20090 Rodano MI
(Italy)

(Manufacturer)

6. Sealed Source Model Designation:

The Radiochemical Centre
Amersham, UK
Code NBC 3

Registered with the U.S.N.R.C.
NRC Mo[illegible] # N.B.C.

7. Nuclide: ^{63}Ni

Activity: 10 mCi

8. Leak Test Frequency: Every six months

9. Principal Use: Ion Generators, Chromatography

10. Custon Device: No

B. DESCRIPTIVE DATA

1. Summary description

The ECD-40 (Electron Capture Detector) is a device for quantitative chemical analysis which operates on the principle of gas phase absorption of free electrons by electron capturing molecules. It consists of an ionization chamber with two electrodes. Carrier gas flows through the chamber. The radioactive source (Ni-63) produces free electrons through a gas ionization process. The beta particles emitted by the Ni-63 source ionize the carrier gas (e.g. nitrogen). Secondary electrons and positive ions are formed. Electrons are collected at the anode generating a constant current. When an electron capturing substance passes through the chamber the current is reduced due to the absorption of electrons by this substance. The output signal is proportional to the molecular concentration of that component.

The ECD-40 module is approximately 12 cm x 11 cm x 5 cm. It attaches to the Carlo Erba Gas Chromatographs. The Carlo Erba Gas Chromatograph is a laboratory instrument used only in controlled environments. It is not subjected to field conditions. The ECD-40 is construcred principally of stainless steel. The source is a 10mm x 24mm rectangular nickel foil, electroplated on one side with 10 mCi ^{63}Ni. The source is secured in a

cylindrical configuration within a stainless steel holder and is held rigidly in place with a stainless steel circlip. The cell is contained within the body of the ECD-40 and is not accessible to the user. The source cannot be accessed or removed from the device either deliberately or accidentally without disassembling the device.

There is no on-off mechanism for the source. It remains in a constant position during use and non-use periods.

2. Labeling

a 2.5 cm x 4 cm etched aluminium label is attached to the external surface of the detector module. The label contains the name of the manufacturer (Carlo Erba Strumentazione), the standard radiation symbol, the nuclide and activity (Ni 63-10 mCi) and the words Caution Radioactive Material. (See Appendix A for drawing and accompanying sample label.)

3. Diagram (See Figure 1)

4. Conditions of Normal Use

The ECD-40 is to be used on a gas chromatograph under controlled analytical laboratory conditions. It is not a field device. Therefore, it will not be subject to vibration, impact, puncture, compressive loads, or extremes of cold under normal operating conditions. Users will generally be trained laboratory technicians or researchers. The device has no alternative use. It is specifically designed to be compatible with the

Carlo Erba Gas Chromatographs and can not be used as a component in any other product.

The useful life of the source is dependent on possible chemical contamination by samples being analyzed. (ERBA recommends complete replacement of severely contaminated cells. Cleaning and replacement of radioactive cells are to be done only by authorized personnel.) The prototype ECD has been in use under normal conditions for 10 years without any deterioration in terms of containment of the radioactive source.

The normal operating temperature limit of the ECD-40 is 399°C. The device is automatically shut down when the temperature reaches the 400°C limit.

No deterioration in the cell or stainless steel housing has been observed when subjected to elevated temperatures for extended periods. An NBC 3 source in a sample detector cell was subjected to a temperature of 450°C in nitrogen for 3 days. No measureable decrease in standing current was observed thus indicating no deterioration of source activity under these conditions.

Since the only use for the source is with the ECD-40 and it is an expensive product, it is highly improbable that it could be diverted from its intended use.

5. Supporting Detail

Complete drawings are included as Appendix A.

The section of the User's Manual devoted to the ECD-40 is included as Appendix B.

C. HEALTH AND SAFETY DATA

1. Safety Analysis Summary

The ^{63}Ni source used in the ECD-40 is manufactured by Amersham Radiochemical Center under model number NBC 3. The source is registered with the U.S.N.R.C. and has been approved for sale in the U.S.A. (The U.S.N.R.C. Model Number is N.B.C.)

2. Manufacturing and Distribution Controls

a. Each ECD-40 is subject to a wipe test for removable radioactive contamination and a pneumatic detector (gas) leak test. Each ECD-40 is certified as being (gas) leak free and having less than 0.005 uCi removealbe ^{63}Ni contamination. Such certification accompanies each unit.

In addition, source activity is automatically checked in the operational check of the device since the baseline current is a function of source activity. Any anomaly in baseline current indicating non-standard source activity would be detected during quality control testing.

b. The manufacturer recommends leak testing at six months intervals and requires that such testing be done by an authorized person. Instructions for leak testing are included as Appendix C.

Installation of the ECD-40 is done by the user and is simply as matter of connecting the detector to the base body of the instrument with the control module. Detailed instructions for installation are included

in the User's Manual (See Appendix B). There is no access to the source at any time during installation or relocation.

The external radiation levels from the ECD-40 are very low and are due only to low energy x-rays produced by interaction of the 67 keV ^{63}Ni beta with the source and housing material. The measured exposure rate at the gas exit port was less than 0.1 mR/hour. Therefore, no on-site radiation exposure rate measurements are necessary.

The manufacturer specifies that any maintenance or service operations involving access to the radioactive source must be performed only by authorized licensed personnel and recommends contacting the ERBA service department for any operations involving cleaning or replacement of the radioactive cell. (See Appendix B).

No emergency procedures are specified due to the low risk involved with use of this source.

c. Manufacturers Instructions to Users (See Appendix B)

The distributor (ERBA Instruments, Inc.) will provide each user with a certification that the source has been leak tested within six months of the time of transfer in addition to a certificate of assay for each source.

Instructions for safe usage of the ECD-40 are included in the User's Manual (See Appendix B).

3. Manufacturers Safety Analysis of Device Review

a. Safety analysis

Under normal operating conditions (i.e. analytical laboratory use) the unit is not subjected to vibration, impact, puncture, compressive loads or abrasion. The source is completely contained within a stainless steel housing and is in direct contact only with the carrier gas and sample. Therefore abrasion of the source is unlikely. Vibration, impact, or compressive loads severe enough to dislodge the source, would disrupt the device operation thus the problem would be immediately apparent. The manufacturer is to be contacted in the event of any instrument malfunction associated with the source. The device housing is stainless steel therefore impact or puncture severe enough to break the housing is highly unlikely under normal conditions.

The carrier gas is inert so corrosion of the source is not likely. It is possible that under certain conditions the sample gas could cause chemical contamination of the source. Any such damage to the source, severe contamination or corrosion, would be apparent due to impaired operating characteristics. The manufacturer recommends return of the device for source replacement or cleaning under these conditions.

b. Prototype testing and evaluation

1) Source

The ^{63}Ni source manufactured by Amersham Radiochemical Centre (Model NBC 3) has been registered with the U.S.N.R.C. and approved for sale in the U.S.

2) The ECD-40 is strictly a laboratory device and will not be subject to harsh field conditions under normal use.

Aging is the major factor affecting source integrity. Ni foil when subjected to high temperatures, will become flexible; to keep the source rigidly in the source holder a spring circlip of stainless steel is used. When subjected to 450°C for three days, no loss of tension was observed. The source can never reach a temperature of 450°c.

The prototype ECD has been in use under normal conditions in Carlo Erba for ten years without any deterioration in terms of containment of the radioactive source.

Under normal working conditions, the limiting factor for the life of the detector is the contamination on the source from sample injection.

Faults in the heating and sensing elements in the ECD-40 will not affect the containment of the source.

Temperature "run-a-way" on the gas chromatograph detector block (raising temperture to 450°C) has been shown not to affect the ECD-40 where the temperature of the source is within the

400^{o}C limit.

No deterioration in the cell or housing (stainless steel) has been observed when subjected to elevated temperatures for extended periods.

3) The device contains a ^{63}Ni source. ^{63}Ni is a pure beta emitter. The only x-rays emitted from the source are due to bremsstrahlung and Ni K x-rays. The bremsstrahlung photon energies are low and range up to 67 keV. Less than 0.2% of the energy is emitted as bremsstrahlung. The Ni K x-rays have an energy of 10 keV. Less than one K x-ray is emitter per 100 disintegrations. All x-rays emitted from the ^{63}Ni source are very low energy and are absorbed in minimal shielding thickness. Under normal operation there is no direct unshielded path between the source and the exterior of the device. Therefore, radiation exposure measurements of the devices after installation by the user are unnecessary. A measurement taken on the ECD-40 at the gas exit under standard working conditions indicated less than 0.1 mrem/hour (Ratemeter Berthold TOL/D).

c. Estimated radiation dose

1) The ECD-40 can be safely operated by persons not having training in radiation protection since the source is completely enclosed and inaccessible under normal conditions and exposure rates from the device are low.

2) Estimated radiation dose under normal conditions -

The measured radiation dose at the gas exit point was less than 0.1 mrem/hour. (This is the point of maximum exposure since the degree of shielding is least.) It is improbable that anyone would spend a significant amount of time at that point. The gas exit is approximately 7 cm from the source. Assuming the measurement was made with the sensitive volume of the radiation detector at 10 cm from the gas exit, the dose rate at 1 meter from the gas exit would be approximately 0.0025 mrem/hour. An individual seated at that location 2000 hours per year would receive a radiation dose of about 5 mrem. This is 1% of the allowed annual dose of 500 mrem. It is unlikely that any individual would ever be in that close proximity to the device for the entire working day.

No internal dose commitment is expected under normal conditions.

3) Estimated radiation dose under accident conditions

a) External radiation dose

The only significant external radiation dose would be due to Ni K x-rays and bremsstrahlung from the 67 kev beta. The maximum range of the beta is 6.3 mg/cm^2 thus it would not penetrate the outer layer of dead skin cells.

Calculated exposure rate at 25 cm from a 10 mCi ^{63}Ni source:

Exposure rate from the 10 keV Ni K x-ray emitted in 0.1% of the disintegrations:

Mass absorption coefficient (10 keV in air) = 4.66 cm^2/g

(Radiological Health Handbook, 1970)

$$\frac{10 \text{ kev} \times 0.001 \times 1.6 \times 10^{-9} \frac{\text{erg}}{\text{kev}} \times 10 \text{ mCi} \times 2.2 \times 10^{9} \frac{\text{d}}{\text{m-mCi}}}{4 \ (25 \text{ cm})^2} \times \frac{60 \text{ m}}{\text{h}} \times \frac{4.66 \text{ cm}^2}{\text{g}} \times \frac{\text{g - R}}{87 \text{ erg}} = 0.00014 \text{ R/hr} = 0.14 \text{ mR/hr}$$

Exposure rate due to bremsstrahlung:

Fraction of energy converted to bremsstrahlung = $\frac{EZ}{800}$

E = maximum energy of beta = 67 keV = 0.067 MeV

Z = mean atomic number of source material and shielding = 27

$$F = \frac{(0.06/)(27)}{800} = 0.002$$

Assume mean bremsstrahlung photon energy is 10 keV

mass absorption coefficient = 4.66 cm^2/g

Calculated exposure rate due to bremsstrahlung:

$$\frac{67 \text{ keV} \times 0.002 \times 1.6 \times 10^{-9} \text{ erg} \times 10 \text{ mCi}}{4 \quad (25 \text{ cm})^2} \times \frac{2.2 \times 10^9 \text{ d}}{\text{keV} \qquad \text{m-mCi}}$$

$$\times \frac{60 \text{ m} \times 4.66 \text{ cm}^2 \quad \text{g-R}}{\text{h} \qquad \text{g} \quad 87 \text{ erg}} = 1.9 \text{ mR/hr}$$

Total calculated exposure rate at 25 cm from 10 mCi of ^{63}Ni:

1.9 mR/hr + 0.14 mR/hr = 2.0 mR/hr

Surface dose rate = 2.0 mR/h x 0.93 mrem/mR = 1.9 mrem/hr

If a source were deliberately removed from the housing the maximum dose rate at 25 cm would be 1.9 mrem/hr.

It is unlikely that any individual would remain at a distance of 25 cm from the source for more than 5 minutes while handling it.

$$\text{Total dose} = \frac{1.9 \text{ mrem}}{\text{hr}} \times \frac{5 \text{ m- hr}}{60 \text{ m}} = 0.16 \text{ mrem}$$

The individual's hand might be within 5 cm of the source for that same period of time.

$$\text{Dose to hands} = \frac{1.9 \text{ mrem}}{\text{hr}} \times \frac{5 \text{ m -hr}}{60 \text{ m}} \times \frac{(25 \text{ cm})^2}{(5 \text{ cm})^2} = 4 \text{ mrem}$$

A "worst case" situation could occur if a technician placed the source on a lab bench and it remained there indefinitely.

Assume the individual works at 1 meter from the source 2000 hours/year.

$$\text{Annual dose} = \frac{1.9 \text{ mrem}}{\text{hr}} \times \frac{2000 \text{ h}}{\text{yr}} \times \frac{(25 \text{ cm})^2}{(100 \text{ cm})^2} = 240 \text{ mrem/yr}$$

In no case would an individual exceed the limit for normal operation.

It is highly unlikely that the device will be handled that carelessly due to its considerable expense.

b) Internal radiation dose commitment under accident conditions

(1) Ingestion

The worst possible case of radiation dose commitment due to ingestion would occur if by some means an individual were to ingest an entire source. The probability of this occurring is truly negligible. However, even in this case, the dose commitment would not exceed the 15 rem limit for accident conditions.

The Annual Limit of Intake for ^{63}Ni by ingestion (occupational dose) is 3×10^8 Bq. (ICRP 30)[1]. The dose commitment associated with that intake is 5 rem (weighted committed dose equivalent).

1. International Commission on Radiation Protection. "Limits of Intakes of Radionuclides by Workers", 1979 Pergamon Press.

Weighted committed dose equivalent from ingestion of 10 mCi ^{63}Ni:

$$\frac{10\ mCi \times 3.7 \times 10^{7}\ Bq}{mCi} \times \frac{5\ rem}{3 \times 10^{8}\ Bq} = 6\ rem$$

(2) Ingestion could also occur if an individual disregards the manufacturers instructions and removes the source or touches it. Due to the fact that this is an electroplated source it is possible that a small amount of the ^{63}Ni could be transferred to the technicians hand. It is unlikely that more than 1% of the total source activity could be ingested in this manner. Thus the weighted committed dose equivalent would be approximately 60 mrem, well below the 500 mrem/yr limit for normal operation and the 15 rem limit for accidental conditions.

b) Inhalation

Interna' dose commitment due to inhalation could only occur under conditions of fire where the source may be vaporized or under conditions of gradual loss of source activity to the carrier gas vented into the laboratory. It is highly unlikely that a fire could occur of such intensity to vaporize the source while people remain in the area. However, this calculation is presented as a "worst case" situation.

(1) Fire in a laboratory containing the ECD-40

Assume:

Instantaneous vaporization of the ^{63}Ni source

Laboratory dimensions = 10 m x 10 m x 2.3 m

Volume = 2.3×10^2 m^2

Occupants remain in the room for five minutes

Breathing rate = 0.104 m^3/5 minutes

$$\text{Total intake} = \frac{3.7 \times 10^8 \text{Bq}}{2.3 \times 10^2 \text{ m}^3} \times 0.104 \text{ m}^3 = 1.7 \times 10^5 \text{ Bq}$$

The Annual Limit of Intake for ^{63}Ni in the vapor state by inhalation = 3×10^7 Bq (ICRP 30)

$$1.7 \times 10^5 \text{ Bq} \times \frac{5 \text{ rem}}{3 \times 10^7 \text{ Bq}} = 0.028 \text{ rem} = 28 \text{ mrem}$$

(2) A more reasonable accident could be undetected vaporization of the source over a period of time

Assume complete vaporization of the source in a 24 hour period.

$$\text{Equilibrium room concentration} = \frac{3.7 \times 10^8 \text{ Bq}}{2.3 \times 10^2 \text{m}^3 \times 24 \text{ hr} \times \text{EX}}$$

Assume air exchange rate (EX) to be 0.5/hr

Equilibrium room concentration = 1.3×10^5 Bq/m^3

Assume worker remains in the area for an 8 hour working day with a daily breathing rate of 1.2 m^3.

Weighted committed dose equivalent:

$$\frac{1.3 \times 10^5 \text{ Bq} \times 1.2 \text{ m}^3}{\text{m}^3} \times \frac{5 \text{ rem}}{3 \times 10^7 \text{ Bq}} = 22 \text{ mrem}$$

Since the proper operation of the unit is dependent on the source strength remaining constant it is highly unlikely that loss of source activity over a short period of time could occur undetected.

(3) Undetected leakage of source over a full year.

$$\text{Equilibrium concentration} = \frac{3.7 \times 10^8 \text{ Bq} \quad \text{hr}}{8.8 \times 10^3 \text{ hr} \times 230 \text{ m}^3 \times 0.5}$$

$$= 3.7 \times 10^2 \text{ Bq/m}^3$$

The Derived air concentration (DAC) for ^{63}Ni in the vaporized state is $1 \times 10^4 Bq/m^3$ (ICRP 30)

Weighted committed dose eqivalent for full time workers in the laboratory:

$$3.7 \times 10^2 \times \frac{5 \text{ rem} \quad \text{m}^3}{\text{yr} \quad 1 \times 10^4 \text{ Bq}} = 185 \text{ mrem/year}$$

The loss of source integrity would be detected due to improper device operation before a significant fraction of the activity is lost.

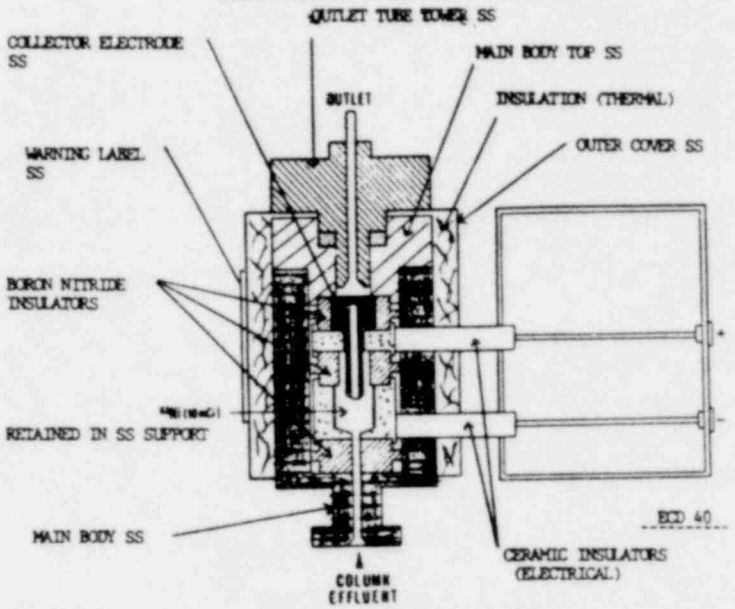
The maximum estimated dose due to inhalation under accidental conditions would be 185 mrem, approximately 1% of the appropriate limit.

There are no accidental conditions that can be postulated which would result in a radiation dose to any individual which would exceed the 15 rem limit.

The are no normal operating conditions which could result in a dose to any individual greater than 500 mrem/year.

APPENDIX "A"

Figure 1: ERBA Instruments, Inc.



SS- Stainless steel to international specification #A151304

ECD-40 MAJOR COMPONENTS, SPECIFICATIONS AND MANUFACTURERS

1. Radioactive source - ^{63}Ni, code NBC3, 10mCi supplied by The Radiochemical Center, Amersham, UK, imported into Italy for manufacture by Italian subsidiary PRODOTTI GIANNI, MILANO.

2. Detector body & cell - Manufactured by Carlo Erba from stainless steel A151304.

3. ECD cell insulators - Boron Nitride manufactured by Union Carbide Corp., USA.

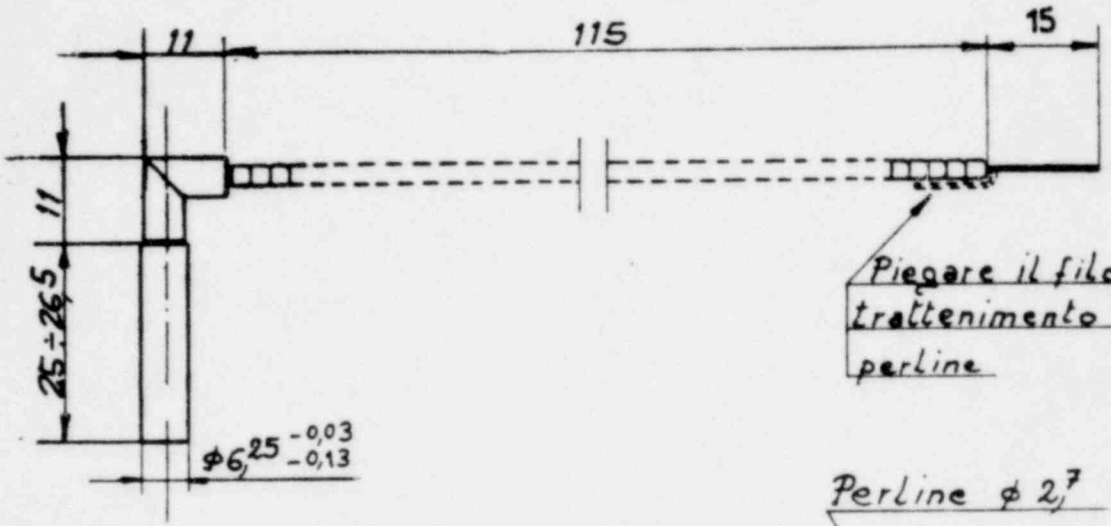
4. Thermal insulation Fibrefrase type 970JH, manufactured by Carborumdum (T.R. SpA Milan).

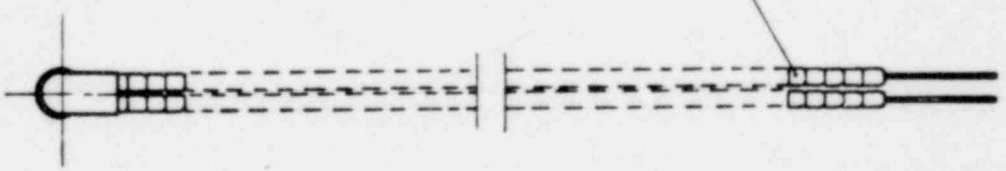
5. Temperature sensor - type PE 100 G 2025 100 ohm glass range-220 -- +550°C manufactured by Heraeus, Germany.

6. Heaters - 2x25W, 24V RICA SpA (Treviso)

7. Electrode insulator - Dugussit A123 code 122110050, manufactured by FRIEDRICHSFELD, MANNHEIN, GERMANY

apice	modifiche	data	firma
2	Aumentato altezza isolatore a 90° era 7x1.	8-2-88	





RISCALDATORE CORAZZATO
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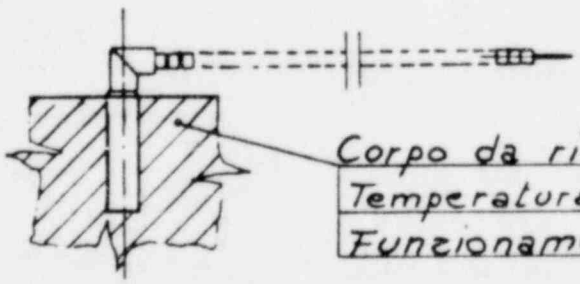
- 4 GEN. 1985

APPLICAZIONE



Corpo da riscaldare - Mat. Aisi 304
Temperatura del corpo - 400°c
Funzionamento - Continuo

pos.	denominazione	pezzi	materiale	note

CARLO ERBA STRUMENTAZIONE

disegno N. 618.001.023

RIVELATORE ECD 40
Riscaldatore

modifica apice 2
dis. Cattaneo data 13-5-87
visto Mandelli scala 1:1

tolleranze per le quote senza indicazione di tolleranza

da 0 a 6 mm	±0,1	±0,2	±0,3	±0,5	±0,8	±1° 30'
da 6,01 a 30 mm	±0,2	±0,3	±0,5	±0,8	±1,2	±1°
da 30,01 a 120 mm	±0,3	±0,5	±0,8	±1,2	±2	±0° 30'
da 120,01 a 315 mm	±0,5	±0,8	±1,2	±2	±3	±0° 20'
oltre 315 mm	±0,8	±1,2	±2	±3	±5	±0° 15'

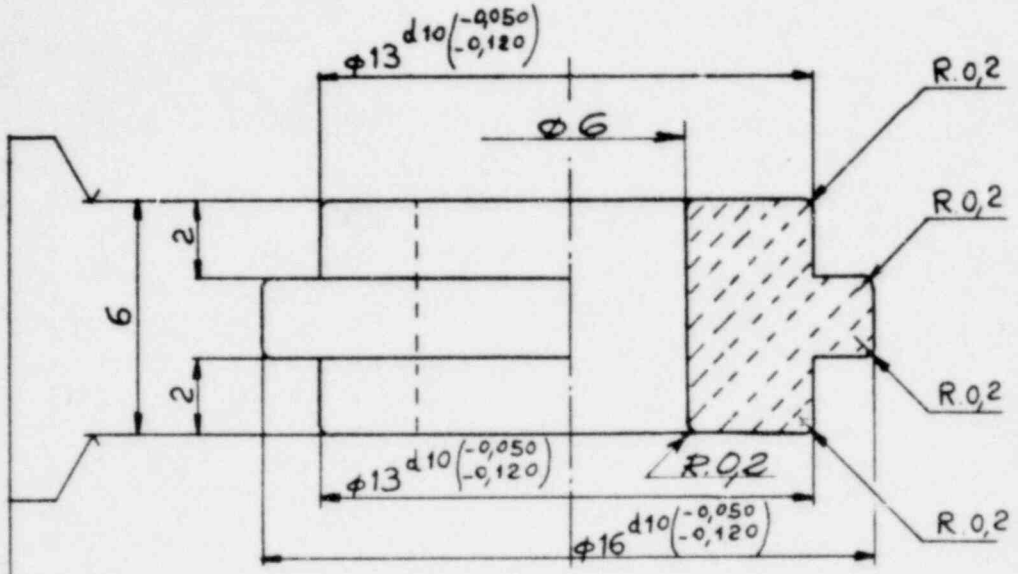
rugosità in μ
- ∇ Ra 6
- $\nabla\nabla$ Ra 0,8
- $\nabla\nabla\nabla$ Ra 0,2
- $\nabla\nabla\nabla\nabla$ Ra 0,025

toller. filettature
- madrevite 6H
- vite 6g

apice	modifiche	data	firma

rugosità in μ	
▽	Ra 4
▽▽	Ra 0,8
▽▽▽	Ra 0,2
▽▽▽▽	Ra 0,025

tolleranze filettature	
madrevite	6H
vite	6g



Superfici perfettamente parallele Ra 0,2

Lavorazione eseguita al tornio senza nessun liquido refrigerante, evitare il contatto con le mani sporche.

Mat. BORON NITRIDE
(Union Carbide)

UFF. [illegible]
DATA - 4 GEN 19[illegible]5
FIRMA .....

pos.	denominazione	pezzi	materiale	note

scostamenti per le quote senza indicazione di tolleranza					
da 0,1 mm a 6 mm	±0,2	±0,1	±0,05		
da 6,01 mm a 30 mm	±0,5	±0,2	±0,1		
da 30,01 mm a 120 mm	±0,8	±0,3	±0,15		
da 120,01 mm a 315 mm	±1,2	±0,8	±0,2		
oltre 315 mm	±2	±0,8	±0,3		
angolari	±1°	±0°30'	±0°20'		

CARLO ERBA STRUMENTAZIONE

disegno N 618.001.016

modifica apice

dis [illegible] data 12/10/81

visto [illegible] scala 5:1

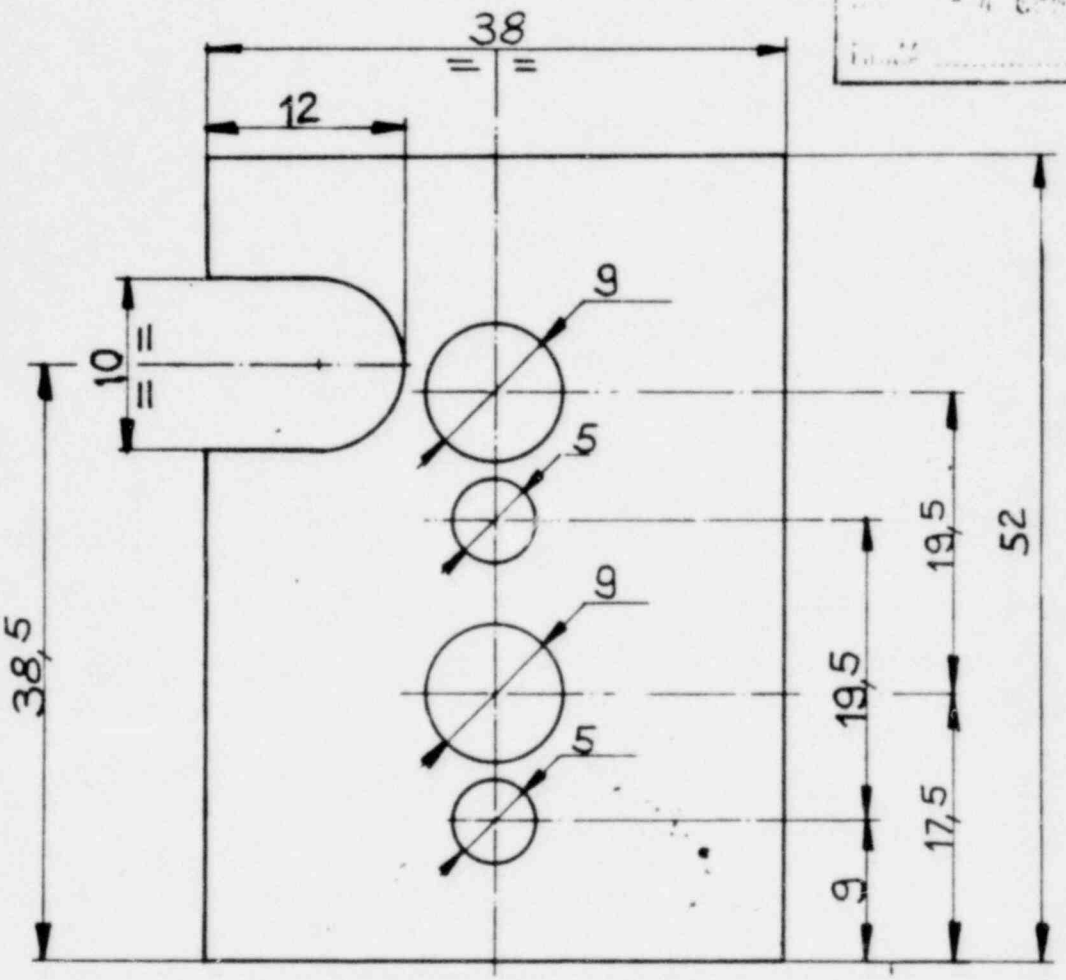
RIVELATORE ECD 40
Isolatore superiore

apice	modifiche	data	firma

rugosità in µ	
▽	Ra 4
▽▽	Ra 0,8
▽▽▽	Ra 0,2
▽▽▽▽	Ra 0,025

toller. filettature	
madrevite	6H
vite	6g

UFF. [illegible] - 4 GEN. 1985



N.B. Tutti i fori devono essere passanti

Mat. Foglio isolante FIBERFRAX tipo 970 JH
senza legante organico spessore: 3,2 (T.R.)

pos.	denominazione	pezzi	materiale	note

oltre 0 mm a 6 mm	oltre 6 mm a 30 mm	oltre 30 mm a 120 mm	oltre 120 mm a 315 mm	oltre 315 mm	angolari
± 0,2	± 0,5	± 0,8	± 1,2	± 2	± 1°
± 0,1	± 0,2	± 0,3	± 0,5	± 0,8	± 0° 30'
± 0,05	± 0,1	± 0,15	± 0,2	± 0,3	± 0° 20'

escludendo per le quote senza indicazione di tolleranza

CARLO ERBA STRUMENTAZIONE

disegno N. 618.001.019

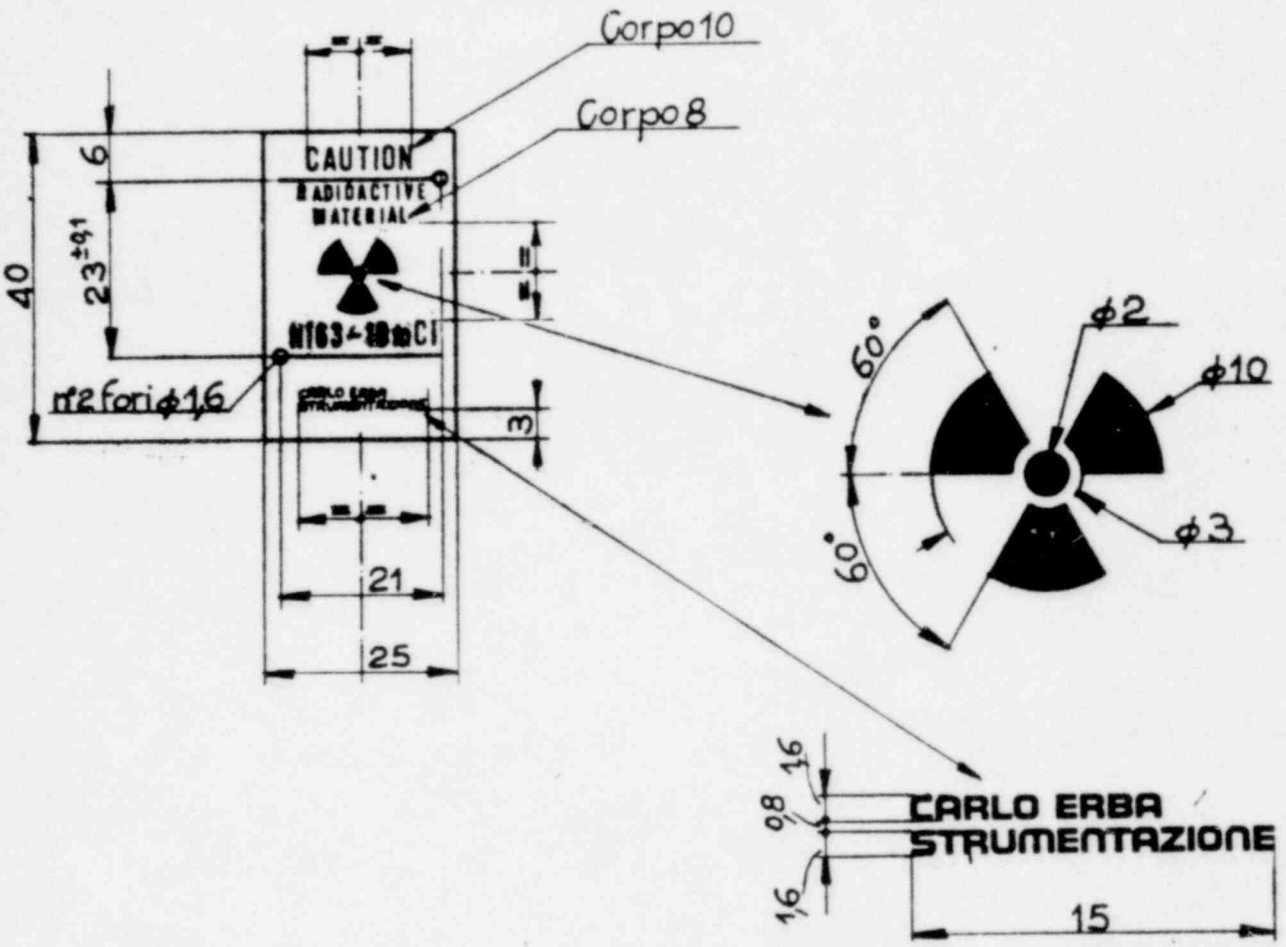
RIVELATORE ECD 40
Isolante termico A

modifica apice							
dis. ABani	data 2/3/81						
visto	scala 2:1						

apice	modifiche	data	firma

tolleranze lavorazioni	
rugosità in μ	
▽ Ra 4	
▽▽ Ra 0,8	
▽▽▽ Ra 0,2	
▽▽▽▽ Ra 0,025	

toller. filettature
madrevite 6H
vite 6g



Diciture e simbolo colore NERO
su fondo colore giallo. (I colori devono rimanere inalterati alla temperatura di 200°)

Microgramma stretto neretto Corpo8 (h 2,3 m/m

NB. Per le diciture delle targhe [illegible]

Microgramma stretto neretto [illegible]

" " "

" " "

Corsetto largo neretto

I seguenti dati, [illegible]
della ditta STYLGRAF (MI)

URF. [illegible]ZIONE
DATA - 4 GEN. 1985
FIRMA .....

Mat. lastra ALLUMINIO sp. 0,5

pos	denominazione	pezzi	materiale	note

accoppiamenti per la quale servono indicazioni di tolleranza	da 0 mm a 6 mm	da 6 mm a 30 mm	da 30 mm a 120 mm	da 120,01 mm a 315 mm	oltre 315 mm	angolari
	±0,2	±0,5	±0,8	±1,2	±2	±1°
	±0,1	±0,2	±0,3	±0,5	±0,8	±0°30'
	±0,05	±0,1	±0,15	±0,2	±0,3	±0°30'

CARLO ERBA STRUMENTAZIONE

disegno N 618.001.110

modifica apice

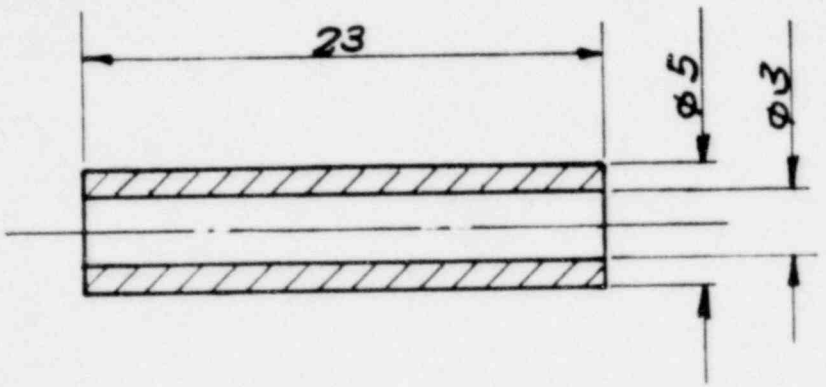
dis Alfani data 4-12-84

visto [illegible] scala 1:1 (3:1)

RIVELATORE A CATTURA DI ELETTRONI ECD 40
Targhetta

apice	modifiche	data	firma
1	Variata cod. era 302.100.07	5/7/82	CF

toller. filettature	rugosità in μ
madrevite 6H	▽ Ra 4
vite 6g	▽▽ Ra 0,8
	▽▽▽ Ra 0,2
	▽▽▽▽ Ra 0,025



Mat. Tubo Allumina pura sinterizzata

(DEGUSSIT AL 23)

cat. FRIEDRICHSFELD cod. N° 122110050

pos	denominazione	pezzi	materiale	note

disegno N 618.001.026

RIVELATORE ECD 40

modifica apice

dis. Mandaglio data 1-7-82

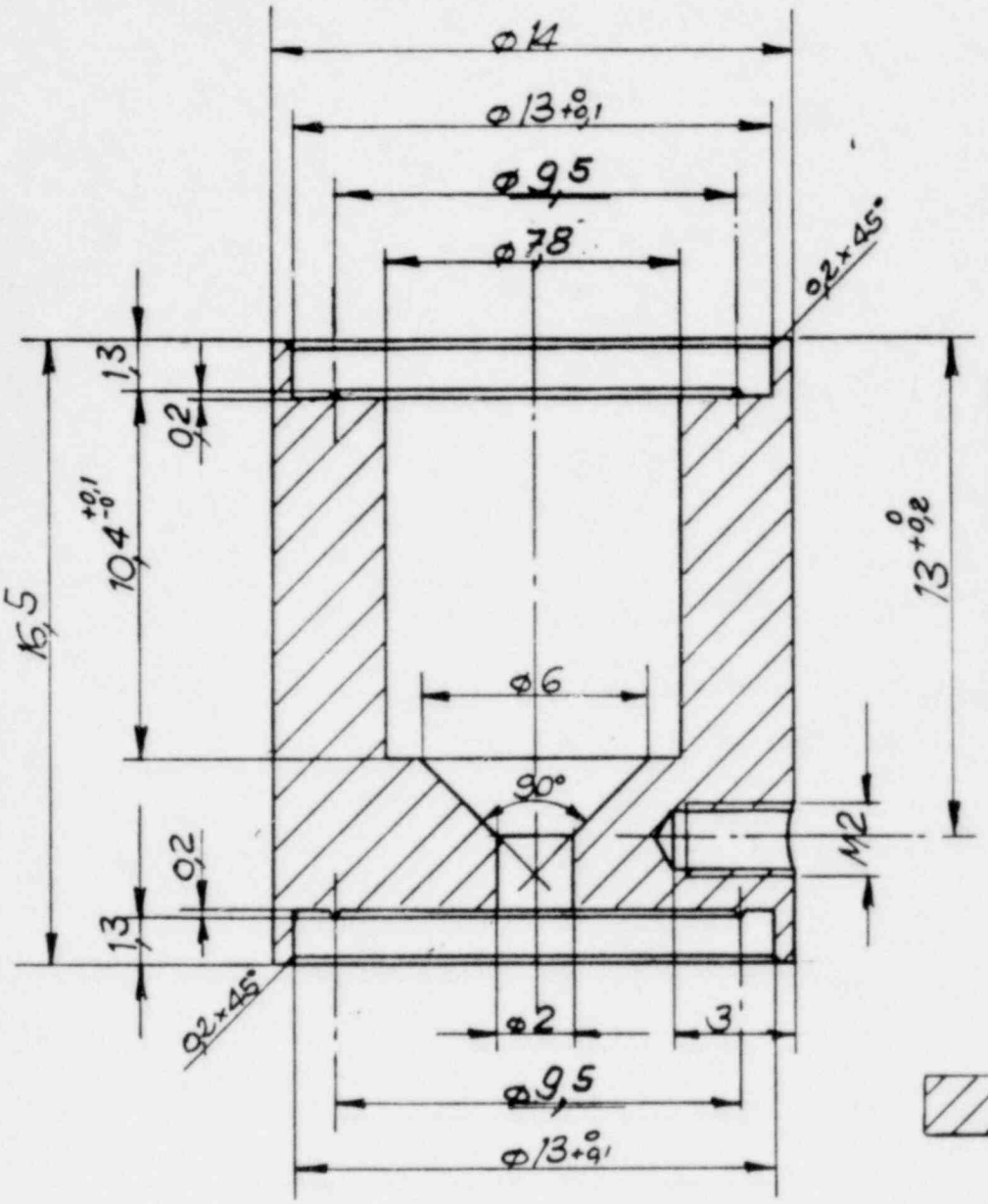
visto scala 3 : 1

accettabili per le quote senza indicazione di tolleranza

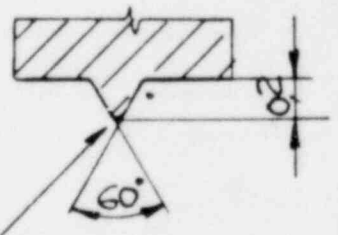
da 0 mm a 6 mm	± 0,2	fino a 30 mm	± 0,5	oltre 30 mm a 120 mm	± 0,8	oltre 120 mm a 315 mm	± 1,2	oltre 315 mm	± 2	angolari	± 1°
oltre 6 mm a 18 mm	± 0,1	oltre 18 mm a 80 mm	± 0,2	oltre 80 mm a 250 mm	± 0,3	oltre 250 mm a 1000 mm	± 0,5	oltre 1000 mm	± 0,8	angolari	± 0°30'
oltre 18 mm a 30 mm	± 0,05	oltre 30 mm a 120 mm	± 0,1	oltre 120 mm a 400 mm	± 0,15	oltre 400 mm a 1600 mm	± 0,2	oltre 1600 mm	± 0,3	angolari	± 0°20'

apice	modifiche	data	firma
	Profondità sede portasorgente portata a 10 era 9,8	28-6-82	
	Profondità sede portasorgente 10,2 era 10.	21-9-82	

rugosità in μ		toller. filettature
▽	Ra 4	madrevite 6H
▽▽	Ra 0,8	vite 6g
▽▽▽	Ra 0,2	
▽▽▽▽	Ra 0,025	



UFF. TECNICO STRUMENTAZIONE
DATA - 4 GEN. 1985
FIRMA



Levigato e senza spigolo tagliente

Mat. X8 Cr Ni 1910 - UNI 6900 (AISI 304)

pos	denominazione	pezzi	materiale	note

scostamenti per le quote senza indicazione di tolleranza

da 0 mm a 6 mm	da 6,01 mm a 30 mm	da 30,01 mm a 120 mm	da 120,01 mm a 315 mm	oltre 315 mm	angolari
± 0,2	± 0,5	± 0,8	± 1,2	± 2	± 1°
± 0,1	± 0,2	± 0,3	± 0,8	± 0,8	± 0° 30'
± 0,05	± 0,1	± 0,15	± 0,2	± 0,3	± 0° 20'

CARLO ERBA STRUMENTAZIONE

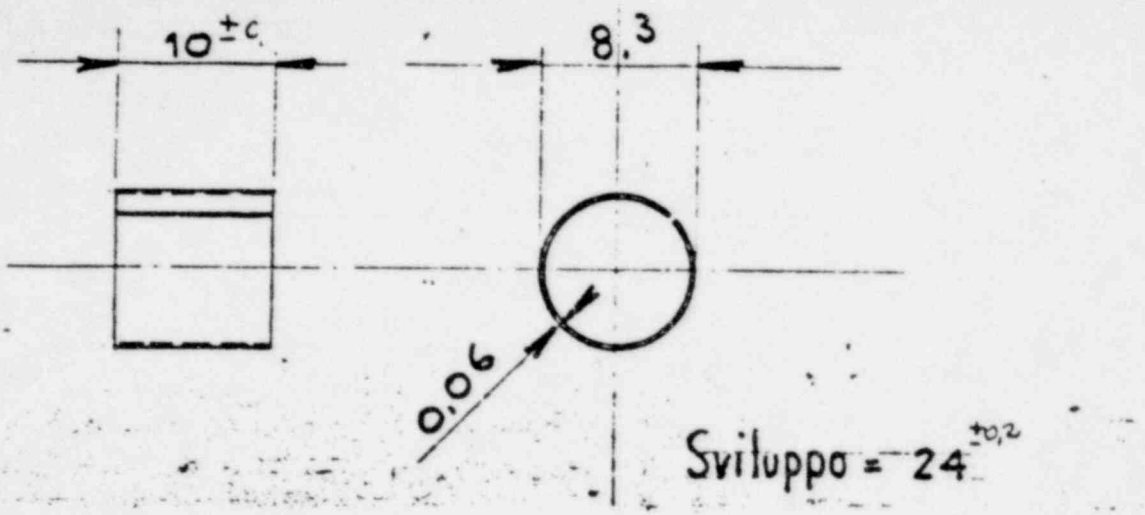
disegno N. 618.001.013

RIVELATORE ECD 40

modifica apice

dis. data 16-2-81

visto scala 5 : 1



MATERIALE DELLA SORGENTE: Ni 63 ELETTRODEPOSITATO SU FOGLIO DI NICHEL.

ATTIVITA' RADIOATTIVA: 10mCi

CODICE DELLA SORGENTE: NBC 3

ESISTE ANCHE DIS. 544/005

CODICE	465	002	00		

1° FOGLIO

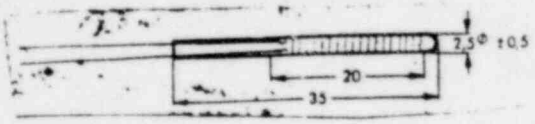
SPECIFICA MATERIALI

N°	FORNITORI
1	PRODOTTI GIANNI (Provenienza: the Radiochemical Center - Amerstham)

N°	PRESCRIZIONI PER IL COLLAUDO

N°	REVISIONI	ESECUTORE	CONTROLLO	APPROVAZ
	Aggiunta tolleranze	[illegible]	9-9-82	

TITOLO: SORGENTE RADIOATTIVA Ni 63	ESECUTORE	CONTROLLO	FOGLIO N 1 SEGUE N



Caratteristiche:
Tipo: 1 PT 100 G 20 25
Resistenza: 1 X 100 Ohm
Isolamento: vetro
Temperatura funzionamento -220 + +550/600°C

1° FOGLIO SPECIFICA MATERIALI

N°	FORNITORI	
1	HERAEUS	1PT 100 G2025

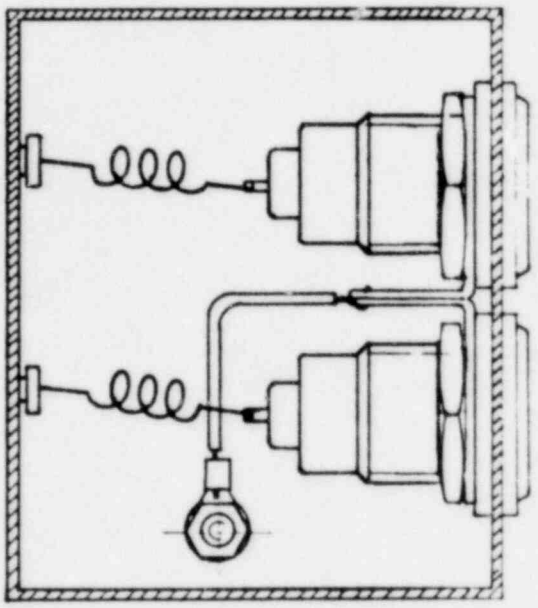
N°	PRESCRIZIONI PER IL COLLAUDO

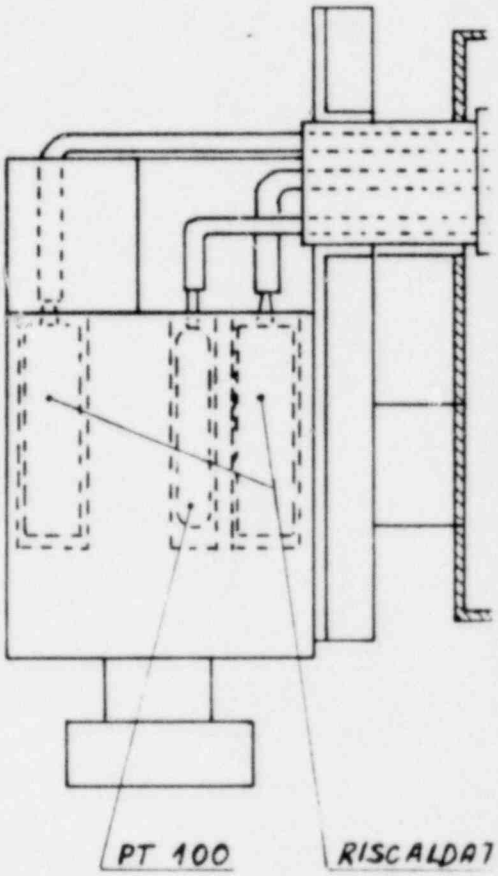
N°	REVISIONI	ESECUTORE	CONTROLLO	APPROVAZ
1	Sost. Codice da 37900100 a 40306800			

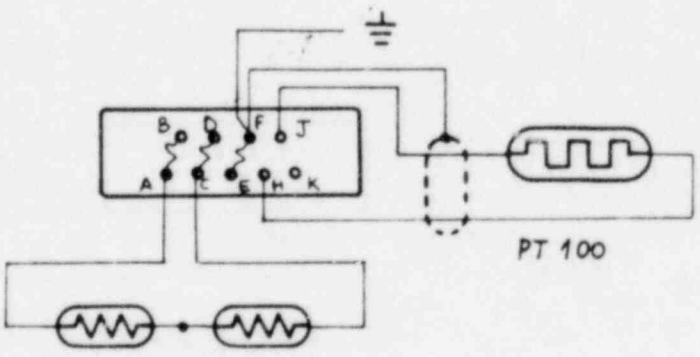
TITOLO: SONDA TERMOMETRICA 1PT100	ESECUTORE	CONTROLLO	FOGLIO N° 1 SEGUE N° —

rugosità in μ	
▽	Ra 4
▽▽	Ra 0,8
▽▽▽	Ra 0,2
▽▽▽▽	Ra 0,025

toller. filettature	
madrevite	6H
vite	6g

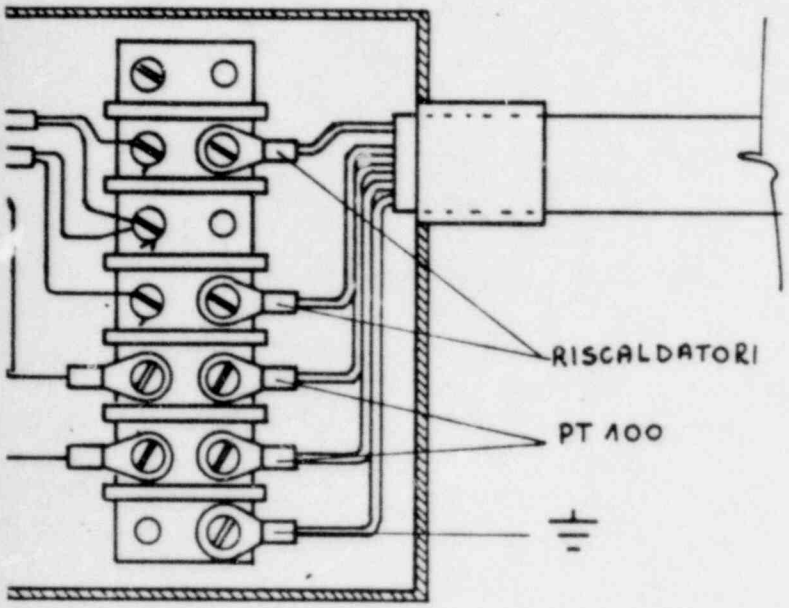






RISCALDATORI
2 × 24V · 25W

grado di precisione	da 0 mm a 6 mm	6,01 mm a 30 mm	30,01 mm a 120 mm	120,01 mm a 315 mm	oltre 315 mm	angolari
grossolano	± 0,2	± 0,5	± 0,8	± 1,2	± 2	± 1°
medio	± 0,1	± 0,2	± 0,3	± 0,8	± 0,8	± 0° 30'
preciso	± 0,05	± 0,1	± 0,15	± 0,2	± 0,3	± 0° 20'



TI
APERTURE
CARD

Also Available as
Aperture Card

RI

UFF. DOCUMENTAZIONE
DATA - 4 GEN 1985
FIRMA .....

8604020341-01

pos	denominazione	pezzi	materiale	note

CARLO ERBA STRUMENTAZIONE

disegno N. SC 204

ECD 40

SCHEMA DI CABLAGGIO

modifica apice

dis. Pantè | data 6-4-83

visto | scala /

sostituisce

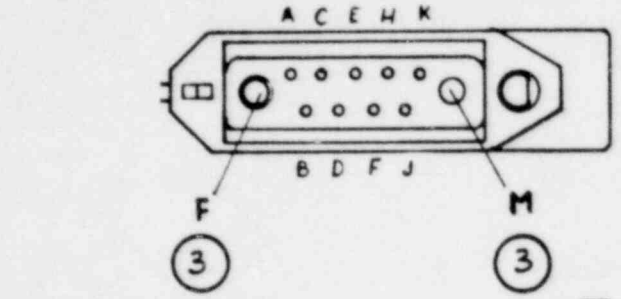
GRUPPO MONTEDISON FARMITALIA CARLO ERBA

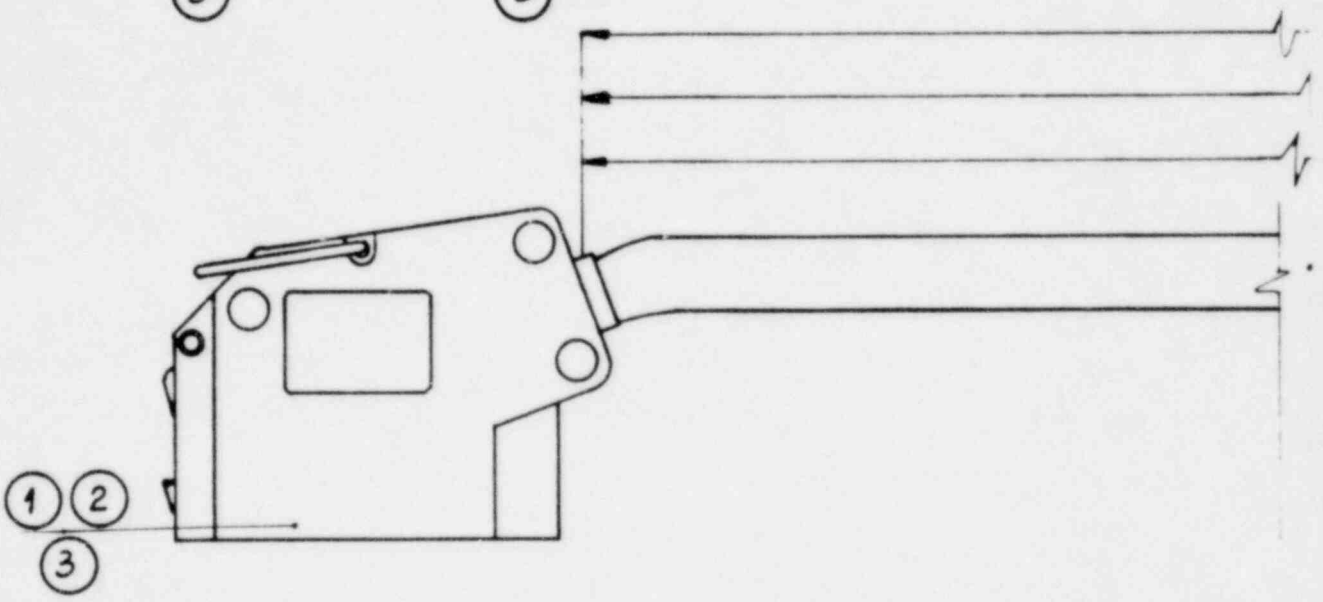
MARCHIO REG. DELLA MONTEDISON S.p.A.

Codice N°

rugosità in μ	
▽	Ra 4
▽▽	Ra 0.8
▽▽▽	Ra 0.2
▽▽▽▽	Ra 0,025

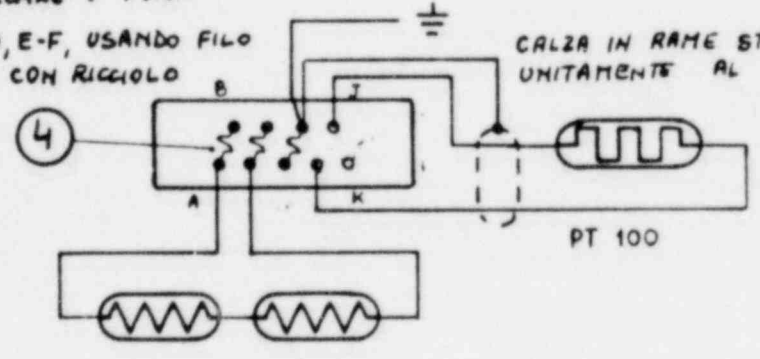
toller. filettature	
madrevite	6H
vite	6g





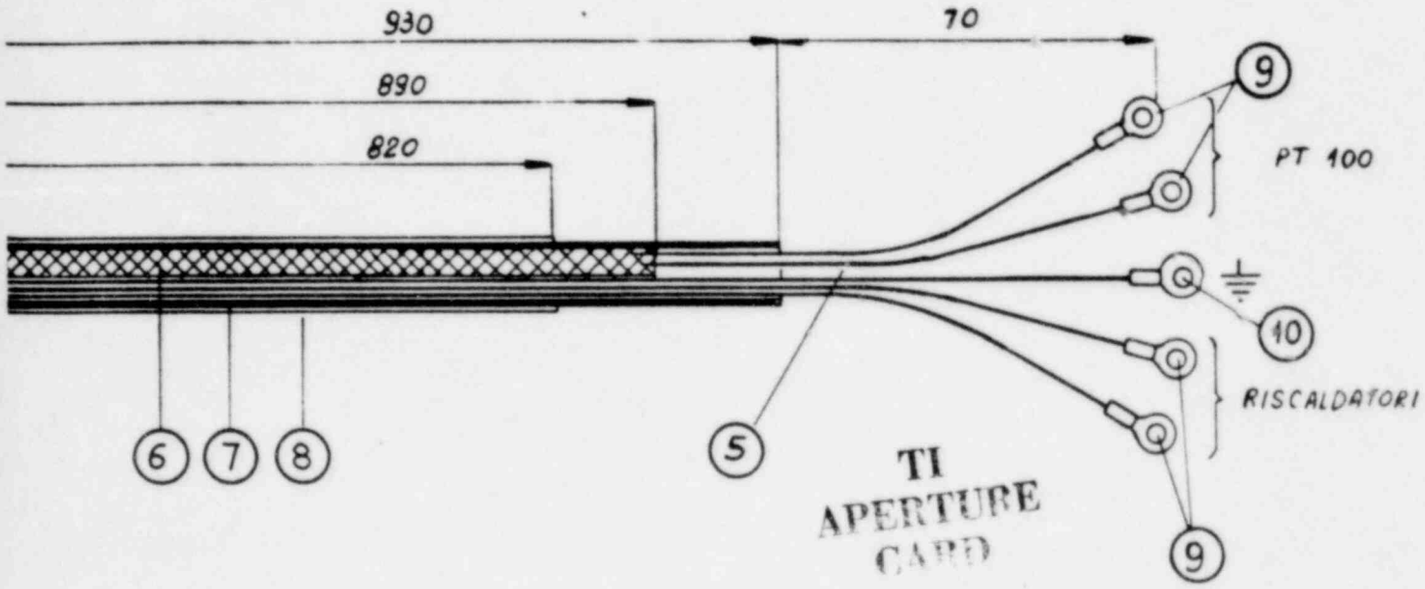
PONTICELLARE I PUNTI
A-B, C-D, E-F, USANDO FILO
DA 0,127 CON RICCIOLO

CALZA IN RAME STAGNATO SALDATA SUL CONNETTORE
UNITAMENTE AL FILO DI TERRA



RISCALDATORI
2 × 24V - 25W

grado di precisione	da 0 mm a 6 mm	da 6,01 mm a 30 mm	da 30,01 mm a 120 mm	da 120,01 mm a 315 mm	oltre 315 mm	angolari
grossolano	±0,2	±0,5	±0,8	±1,2	±2	±1°
medio	±0,1	±0,2	±0,3	±0,8	±0,8	±0°30'
preciso	±0,05	±0,1	±0,15	±0,2	±0,3	±0°20'



Also Available On Aperture Card

8604020341-02

pos.	denominazione	pezzi	materiale	note

CARLO ERBA STRUMENTAZIONE

disegno N. FC 369

CAVO DI COLLEGAMENTO PER ECD 40

ASSIEME

modifica apice

dis. [illegible] data 6-4-83

visto

scala /

sostituisce

GRUPPO MONTEDISON
FARMITALIA CARLO ERBA

MARCHIO REG. DELLA MONTEDISON S.p.A.

Codice N°

APPENDIX "B"

ELECTRON CAPTURE DETECTOR - MODEL ECD-40 - DRAFT

ADDITIONAL INFORMATION FOR U.S. CUSTOMERS.

Please read carefully the ECD-40 manual together with the information detailed below and take appropriate action if required before opening the detector base.

Electron capture detectors must be subjected to a wipe test at least every six months by approved personnel. * Records of these wipe tests must be kept for inspection by the Nuclear Regulatory Commission or the appropriate authority.

While the detector is not in use, it should be kept in a secure location under the direction of a responsible person. The secure location should be locked and display the international radioactivity warning label.

The method used for wipe tests is detailed on the reverse of the original wipe test certificate supplied with the detector.

The source in the ECD-40 has the following characteristics:

Radionuclide	63Ni
Activity	10mCi
Half Life	100 years
Physical State	63Ni electrodeposited on Ni foil

The detector is licensed by the NRC No. XXXX, and records will be kept by Erba Instruments, Inc. of the delivery address of the user.

The detector will not allow emission of radioactive gas when used; safety features incorporated in the detector design will prevent any hazard to the user. However, the operating and safety instructions must be read prior to installation or operation.

* Wipe tests must only be carried out by authorized personnel approved by the local radiation authority. Please contact Erba Instruments, Inc. if in any doubt.

4.1.2

ELECTRON CAPTURE DETECTOR ECD-40 AND CONTROL MODULE ECD-400

INSTRUCTION MANUAL

–TABLE OF CONTENTS–

The Electron Capture Detector ECD-40 can be easily installed on any Carlo Erba chromatograph (Fractovap, Mega Series) and is interchangeable with the other ionisation detectors.

It can operate only when connected to the ECD Control Module-400 since detector and control module are interdependant.

WARNING: The ECD-40 contains a Ni^{63} beta-emitting radioactive source. The radioactive risk associated with the correct use of this detector is practically non-existant. In fact the radioisotope, which is electrically deposited as metal on a nickel foil, is not released by its support at temperatures lower than 450°C. This value can never be reached by the detector whose maximum operating temperature is 399°C and is provided with an overheating safety device. Detector use does not therefore require any particular cautions. However, any maintenance or service operations involving access to the radioactive source must be performed ONLY by authorised CARLO ERBA personnel specifically licensed to handle radioactive material.

We recommend you to contact your local Carlo Erba Service organization for any operations involving cleaning or replacement of the radioactive source.

Alternatively the whole detector can be returned to us for repair.

GENERAL

Operating Principle

The ECD operates on the principle of gas phase absorption of free electrons by electron capturing molecules. Basically it consists of an ionisation chamber with two electrodes. Carrier gas flows through this chamber and an ionising radioactive source produces free electrons through a gas ionisation process. The primary electrons emitted by the radioactive source (β-emission), collide with the molecules of the carrier gas (e.g. nitrogen) and originates an ionisation process with formation of secondary electrons and positive ions (equation 1):

$$\beta + N_2 \rightarrow \beta' + N_2^+ + e \qquad (1)$$

Due to the electric field between the electrodes, the electrons are easily collected at the anode generating a constant current of the order of a few nA, consequently the possibility for the positive ions to recombine with the free electrons is negligible. When an electron capturing substance passes through the detector the electric current is reduced owing to the absorption of electrons by this substance which can be rationalised according to one of the following reactions (equations 2, 3):

$$e + AB \rightarrow \overline{AB} + h\nu \qquad (2)$$

$$e + AB \rightarrow A^{\cdot} + B^- \qquad (3)$$

In (2) an energised negative molecular ion is formed, while in (3) after electron capture the molecule proceeds to dissociate (dissociative capture) into a free radical $A^{\cdot}$ and negative ion B^-. The energy liberated by capture in (2) or the dissociation of the negative molecular ion in (3) is the measure of the electron affinity of the molecule.

The succession of phenomena that determines the response of the detector ends with the neutralisation of the negative ions formed by "capture". Such response then appears as a reduction of the standing current, since at the moment of detection a part of the free electrons is completely removed from the system in equilibrium. The electronic circuitry senses the change in free electron concentration and generates an output signal proportional to the sample molecular concentration.

The sensitivity and selectivity of response of the ECD are determined by, the electron affinity of the substances that enter the ionisation chamber. In the case of organic compounds, the electron affinity depends mainly on the predominant functional group in the molecule, such as halogens, esters, hydroxyls and other oxygenated groups.

The response factor can vary between $1–10^7$ for a low or a high electron capturing substance respectively. These factors are also affected by temperature.

Considering the different responses, it is necessary to calibrate the detector for compounds to be analysed quantitatively. Calibration is possible by injection of standard samples under the same operating conditions.

Table 1 gives the relative sensitivity values for some common substances.

Table 1. ECD relative sensitivity of some compounds.

Substances	Relative sensitivity
Ethane Napthtalene	1
Butanol Acetone Chlorobutane Chlorobenzene	$1–10^2$
1,2-Dichloro ethane Antracene Keto-steroids	$10^2–10^4$
Chloroform Nitrobenzene	$10^4–10^5$
Carbon tetrachloride Dinitrophenol Diethyl fumarate Diethyl oxalate Dihydropyridine	$10^5–10^6$

ECD-40 Operation Modes

The detector can be operated in two pulsed-voltage excitation modes:

Constant Frequency
Constant Current

Constant Frequency Mode (CF)

This mode of operation consists in applying a pulsed voltage of a definite and constant amplitude where the frequency and duration of the pulse is pre-selected. This duration of the pulses is sufficiently long to allow the collection of the very mobile electrons and sufficiently short to enable the heavier and slower negative ions to make a significant contribution to the standing current.

During the application of the pulse the electrons migrate to the anode, consequently their concentration in the cell falls rapidly to zero. During the interval between the pulses, the concentration of electrons gradually returns to the original value, and the phenomena of capture occurs predominantly at this point. When the pulse voltage is under operation, all the electrons not consumed during the relatively long interval between the two short pulses, are collected in the anode and the variation in the resulting cell current produces the detector response.

Under this operation mode it is possible to obtain the best results from a sensitivity point of view.

This method allows the linearity of ECD response to be increased by a factor of 100 compared with the previous mode.

In the constant current mode the variation of the pulse frequency is measured through an electrical feed-back loop circuit which maintain the cell current at a constant reference value by varying the frequency of the applied pulses. When an electron capturing compound enters the detector the pulse frequency is automatically increased to maintain the difference between the cell current and the reference current equal to zero.

The difference between the frequency value when an electron capturing substance is in the cell and the base frequency is computed in an output signal proportional to the sample concentration in the detector.

Fig 4.1.2.1. shows the block diagram of the constant current and constant frequency operation modes.

ECD CONTROL Mod.400 - BLOCK DIAGRAM

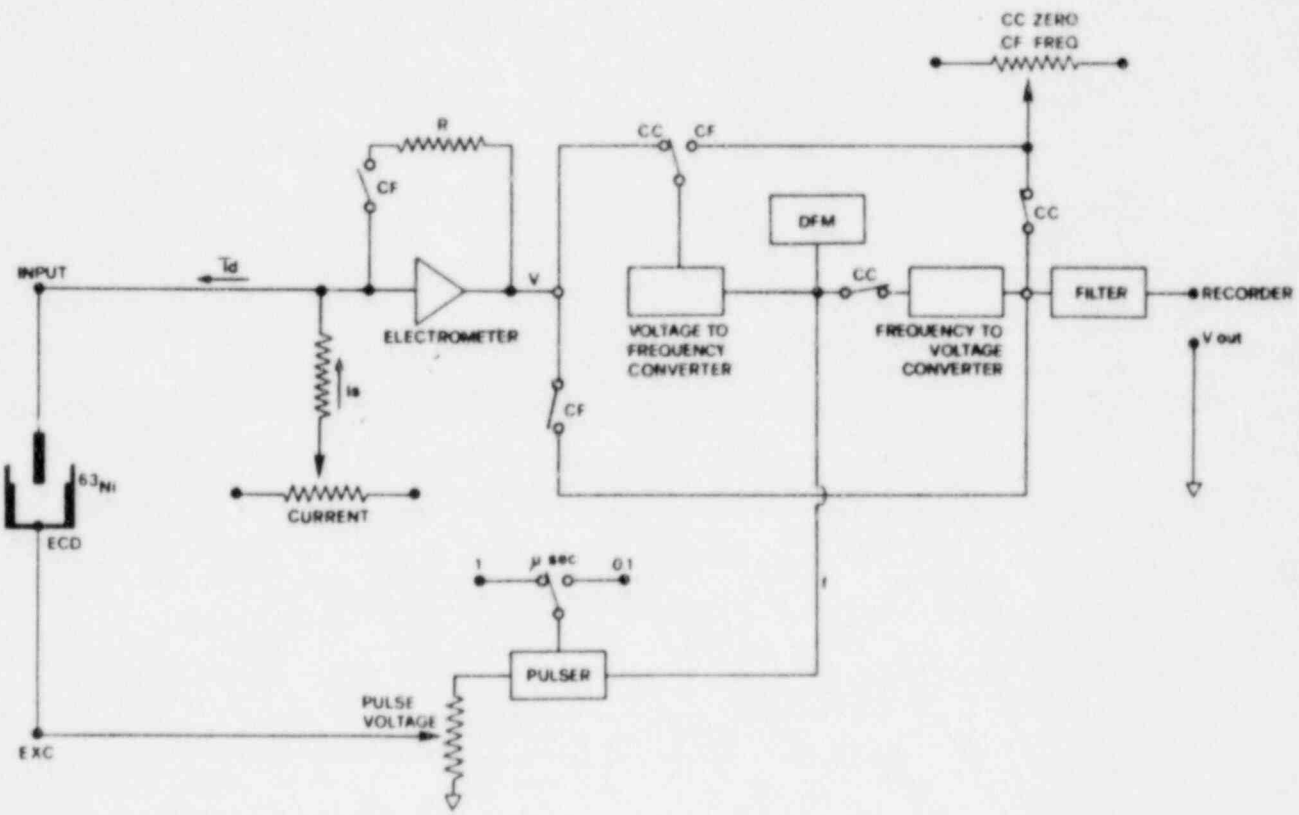


Fig. 4.1.2.1.

Front Panel (Fig 4.1.2.2.)

1. Push button to select Constant Frequency (CF) mode operations.
2. Push button to select Constant Current (CC) mode operations.
3. Dual function knob, used as zero control when CC push button is depressed and as frequency selector when CF push button is depressed.
4. Digital frequency meter giving direct frequency readout (KHz) for both modes of operation.

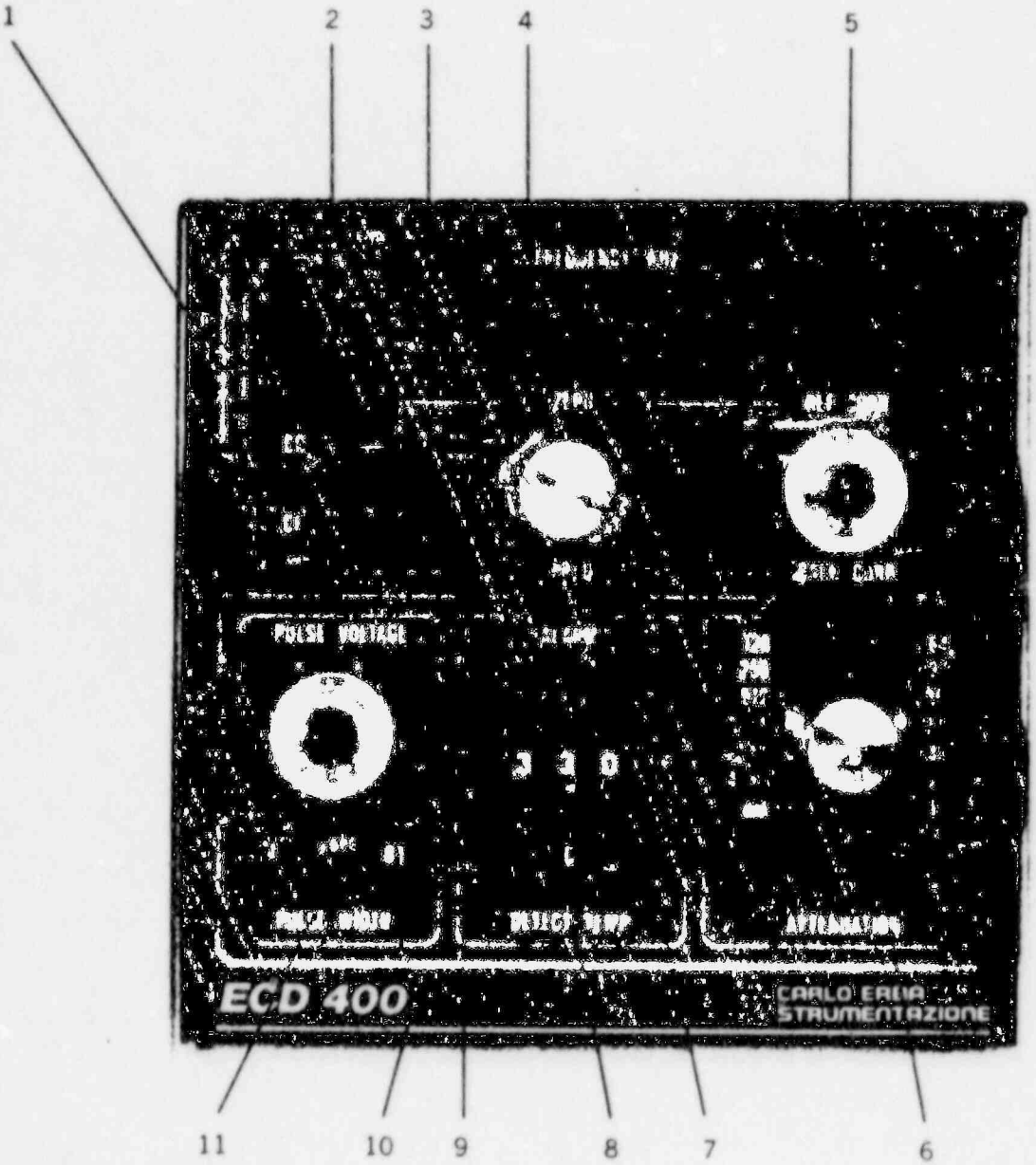


Fig. 4.1.2.2.

5. Five-turn double function potentiometer, used to select the reference current (nA) in CC mode and baseline zero set with standing current readout (nA) in CF mode.
6. Binary steps output attenuator (x1 to x4096).
7. Detector temperature thumbwheel switch (range 0 to 399°C).

8. Detector h ing LED:
   a) blinking when the temperature controller is regulating detector temperature.
   b) full intensity during detector heating phase.
   c) half intensity when detector temperature is higher than the preset one.
9. Detector alarm LED. Light switches from green to red (with subsequent power cut off) in case of detector overheating or when the detector connector (29) is disconnected. To restore original operating conditions, switch module off, and after elimination of the cause of the trouble, switch on again.
10. Five-turn potentiometer to adjust pulse amplitude in the range of 0 to 50V.
11. Pulse width push button selector (μs). Select 1μs when operating with nitrogen or 0.1 μs when using Argon/Methane.

Rear Panel (Fig. 4.1.2.3.)
The rear panel of the ECD-400 Control Module features the following parts:

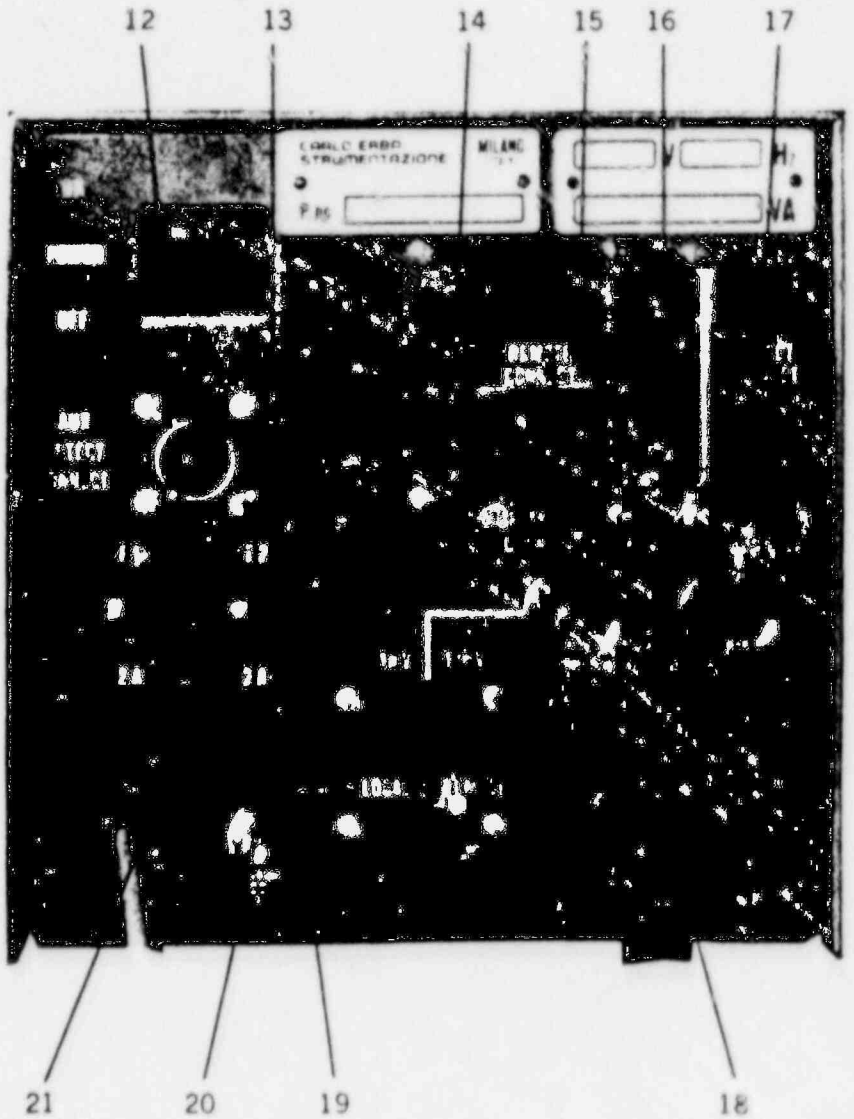


Fig. 4.1.2.3.

14. Output voltage ector (1mV–10mV) for connection to entiometric recorder.
15. Connector for potentiometric recorder.
16. Socket for detector connection (29).
17. Connector for computer-integrator cable. Two different outputs are available 1V–10V and the selection of the required value obeys the pin configuration of the connecting cable as shown in Fig 4.1.2.4. This cable should be grounded to the Integrator only.
18. Detector connection cables (excitation and signal).
19. Fuses (2A).
20. Ground.
21. Mains cord (220V, 50/60 Hz).

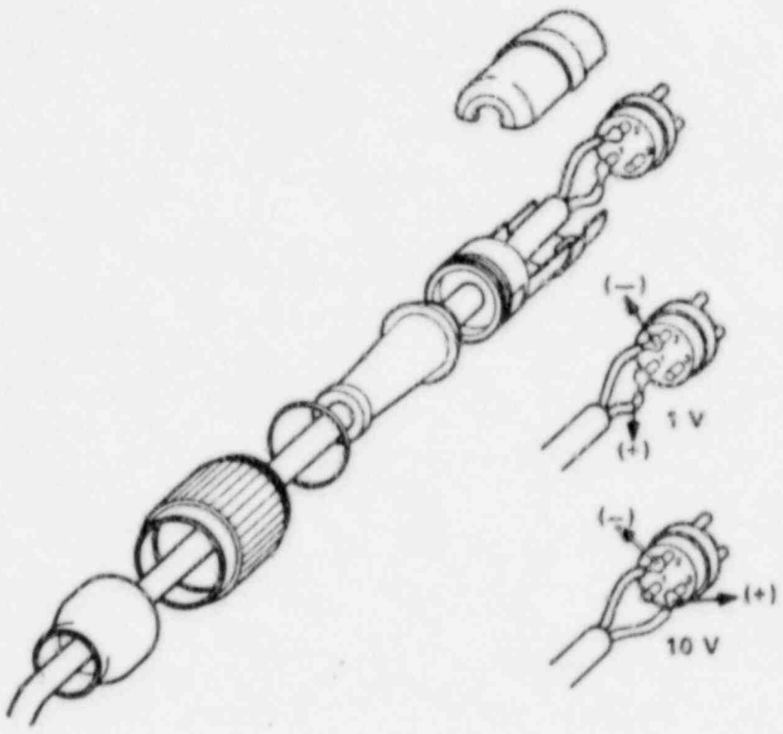


Fig. 4.1.2.4. Connecting cable Computer Integrator to ECD - 400 Control Module.

III DETECTOR ECD-40 DESCRIPTION

The ECD-40 is shown schematically in Fig 4.1.2.5. It comprises of a stainless steel cylindrical holder and a radioactive source. This consists of a film of nickel 63 electro-deposited on a thin metallic foil which acts as the cathode in the ionization cell. Another cylindrically shaped coaxial electrode constitutes the anode. The insulation of the two electrodes from the body of the detector is achieved by means of a high temperature insulator. The detector is heated by two low voltage heaters (48V) controlled by an electronic temperature controller mounted in the ECD Control Module-400.

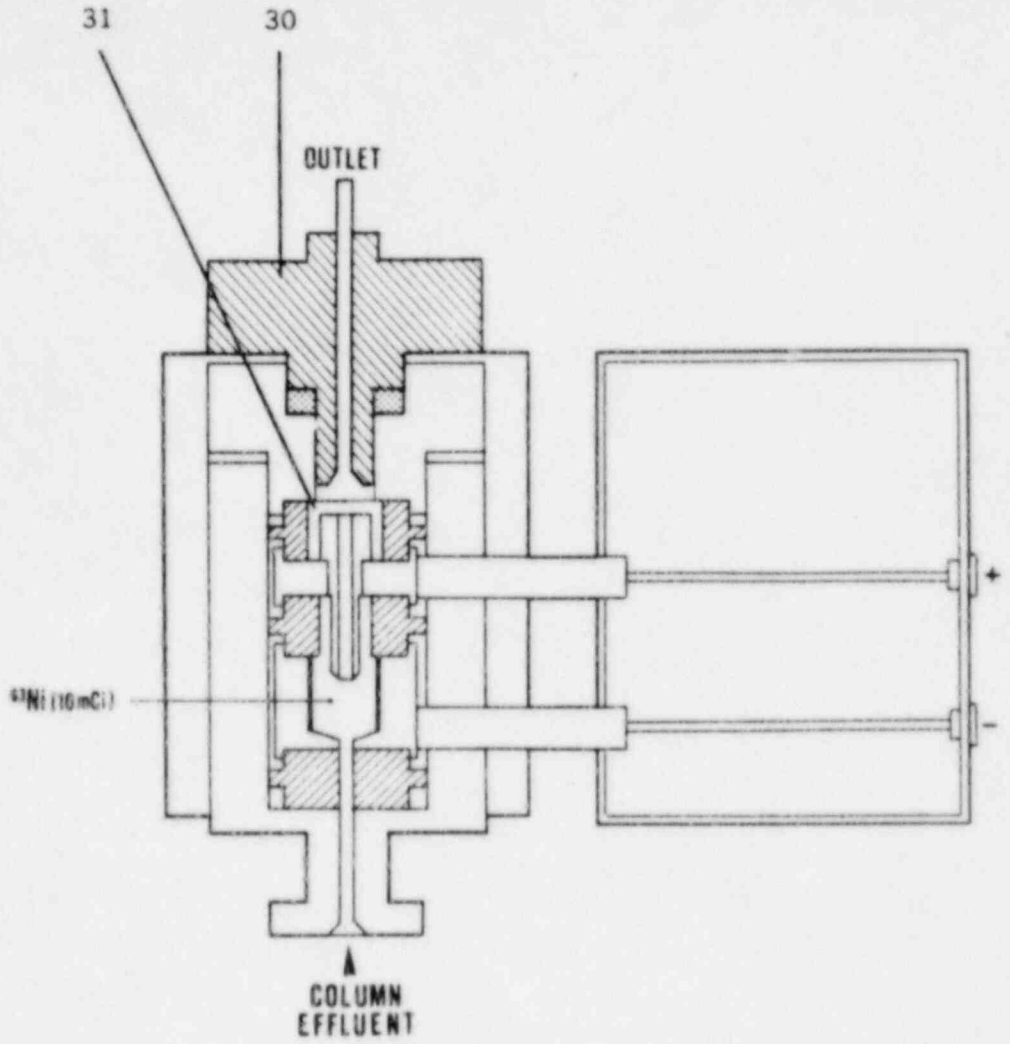


Fig. 4.1.2.5.

The temperature control element consists of a platinum wire sensor connected to the body of the detector.

The connectors to the control module are at the rear of the detector.

Characteristics of the radioactive source:

Radio isotope	: Nickel 63 electro deposited on a nickel support.
Type of radiation	: Low energy beta rays (66 KeV).
Total activity	: 10 mCi.
Half life period	: 100 years.
Radio toxicity	: Moderate (group III).
Physical state	: Solid.

Fig 4.1.2.6. shows the ECD-40 with the standard outfit provided for detector installation on gas chromatograph.

Linear dynamic range : higher than $1:10^4$ (constant current mode).
Minimum detectable amount : less than 0.1 pg of lindane.
Iniosation chamber volume : 400 µl.
Operating temperature limit : 399°C.

ECD 400 Control Module Technical Specifications:

Constant Current Mode.
Reference current : 0 to 5×10^{-9} A, continuously adjustable.
Pulse amplitude : 5 to 50 V pk (negative), continuously adjustable.
Pulse width : 0.1 µs (argon/methane), 1 µs (nitrogen).
Frequency range : 0 to 2.5 MHz @ 0.1 µs, 0 to 500 KHz @ 1 µs.
Computer Output*
Range : 0 to 10 V DC.
Sensitivity : 4µV/Hz@0.1µs, 20µV/Hz@1µs.
Recorder output** : strip chart potentiometric record 10 mV, 0.5 s f.s.
Attenuation : 13 binary steps from 1 to 4096 plus ∞ (shunt).
Sensitivity x 1 : 16 µV/Hz @ 0.1 µs, 80 µV/Hz @ 1 µs.
Constant Frequency Mode
Input range : 0 to 5×10^{-9} A.
Pulse amplitude : 5 to 50 V pk (negative), continuously adjustable.
Pulse width : 0.1 µs (argon/methane)—1 µs (nitrogen).
Frequency setting : 0 to 50 KHz continuously adjustable.
Computer Output*
Range : 0 to 10 V DC.
Sensitivity : $0.1 mV/10^{-12} A$
Recorder output**: strip chart potentiometric recorder 10 mV, 0.5 s f.s.
Attenuation : 13 binary steps from 1 to 4096 plus ∞ (shunt).
Sensitivity x1 : $0.4 mV/10^{-12} A$
*0–1V DC output is also available.
**0–1mV DC output is also available.

IV DETECTOR INSTALLATION

To install the ECD-40 on the gas chromatograph proceed as follows:

1. Replace the flame jet used for FID by the one provided as standard (25) with the ECD-40, using spanners provided. Refer to Fig 4.1.2.6 for identification of parts.
2. Ensure that a PTFE washer (code no. 290 30903) is fitted on the jet.
3. Tighten the detector on the base body by means of the brass nut (code no. 350 01119) (26) and spanner (27) provided.
4. Insert the detector connector into socket (16) at the rear of control module (Fig 4.1.2.2.).
5. Insert the connectors of cables (18) coming from control module, into the corresponding detector sockets (28).
6. Connect the control module to a 1 or 10mV potentiometric recorder through connector (15). Select correct output through switch (14).
7. Plug ECD Control Module 400 into the mains through line cord (21).

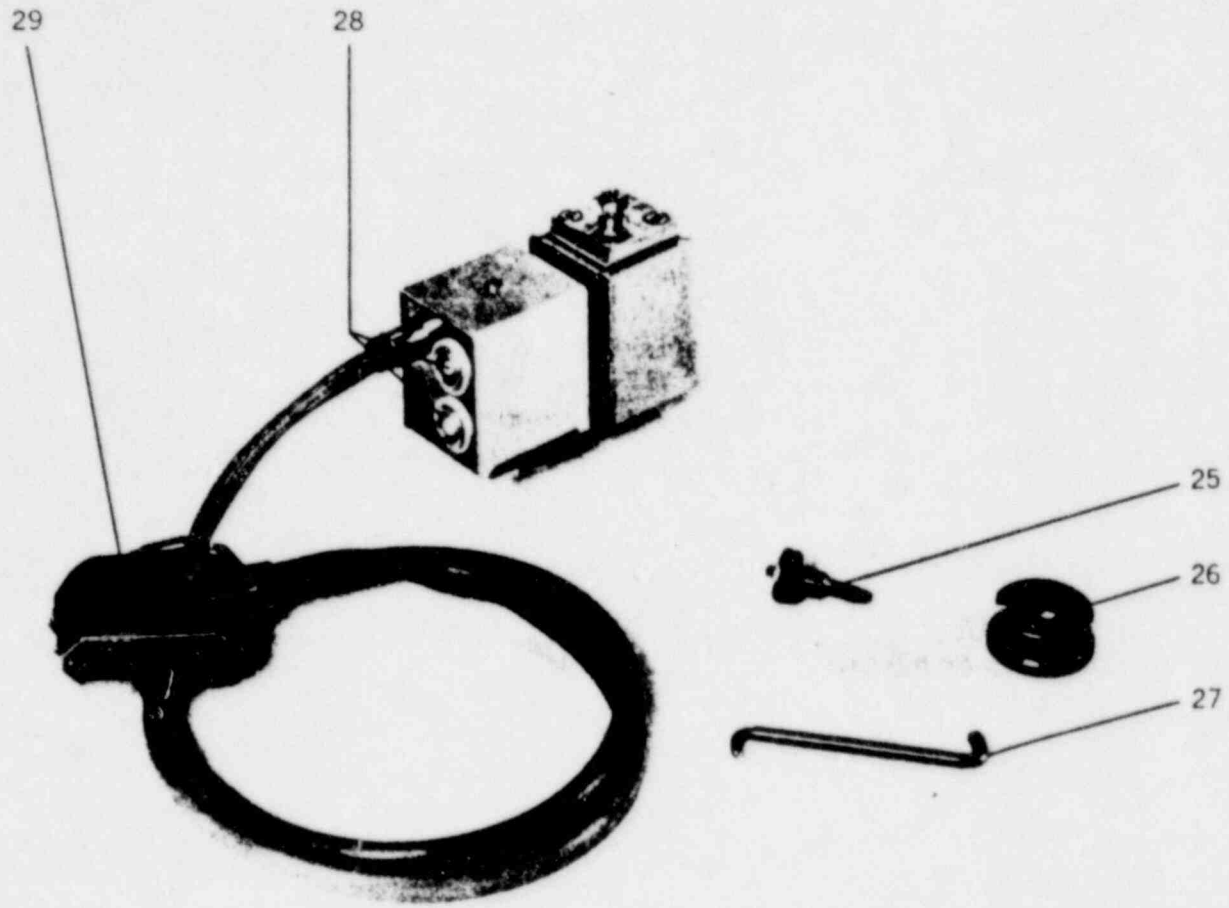


Fig. 4.1.2.6.

Carrier Gas—Make-up Gas

Nitrogen or Argon/5–10% methane should be used. Both gases should be of high purity and must not contain more than 1–2 ppm of oxygen or water vapour as well as other electro capturing compounds which would decrease the concentration of free electrons and the probability of formation of negative ions.

When operating with nitrogen select a 1μs pulse width while 0.1 μs is recommended when using argon/methane.

Argon/methane in fact permits electron collection by the anode at a rate ten times higher than nitrogen.

Comparable results are however obtainable both with nitrogen and argon/methane if the gas chromatographic system is well "clean" (carrier gas, pressure reducer, columns, injection port septum, seals, etc.).

Argon/methane is advisable when the highest degree of linearity is required in the CC mode or when due to a decrease in standing current (due to contaminants in the carrier gas) a high mobility of electrons is necessary to restore correct operating values.

Generally during the detector start-up, an auxiliary gas flow of the same type of the carrier gas should be supplied to the detector. This make-up gas (scavenger) is supplied at a flow rate of 10–30 ml/min through the built in line of the GC or through the unused hydrogen line.

require nitrogen or argon/meth ne as make-up gas at a flow rate of 20 0 ml/min.

*WARNING: When hydrogen is used as the carrier gas, the column oven must be equipped with the HYDROGEN SENSOR DEVICE (code no. 276 05000-50) for safety reasons.

V START UP

Before proceeding to detector heating and start-up, it is necessary to comply with the following:

- Chromatographic column conditioning.
- Choose high temperature stability stationary phases.
- Avoid stationary phases containing halogens or oxygen function groups.

The concentration of stationary phase on the support should be in the range 1 to 5%. Connect only column inlet limb to injection port and leave column outlet limb free in the oven, thus avoiding any possibility of detector base body contamination due to phase leaving the support during conditioning.
Column conditioning requires at least 24h at a temperature of 50°C higher than that used in operations.

During conditioning the carrier gas should flow at the operating rate.

Injection port septum and column ferrules

Prior to installation, it is advisable to heat the septum at 250°C for 24h if possible in an inert gas atmosphere. Otherwise it could slowly release substances such as plasticiers or monomers which could reduce standing current.

Low bleed, high temperature stability ferrules are recommended to avoid cell contamination.

Graphite, graphitised vespel or ceramic loaded PTFE ferrules are suitable for glass columns (including capillary types), while brass ferrules can be used for metal columns, Viton and neoprene o-rings must be avoided.

NOTE: When operating with ECD the column and injector temperature should be kept as low as possible, compatible with the analysis to be run in order to minimize colum and septum bleed.

Gas Chromatograph Start-up

Install column, set desired flow rate and heat detector to a temperature 50°C higher than column, when LED (8) indicates temperature set has been reached (glowing light), switch on GC. It is advisable to leave the instrument under operating conditions for at least 12 hours before injecting the first sample.

NOTE: The sample to be injected should be as clean as possible to avoid detector contamination. It is obvious that halogenated solvents or solvents containing electronegative functional groups must be avoided.

VI CONSTANT CURRENT MODE (CC)

1. Press button (2) and select pulse width by button (11): 1μs for nitrogen and 0.1 μs for argon/methane.
2. Set a voltage of 50V on the "pulse voltage" potentiometer (10). This value may be reduced if the system is ideally clean (see paragraph 4) in order to reduce the excitation level of thermal electrons.
3. Set cell current between 0 and 5 nA by means of the potentiometer (5).

current. As an indication for a current of 1 nA a base frequency lower than 5 KHz should be displayed. Consider also that the higher the preset reference current the higher sensitivity and background noise will be. The best signal to noise ratio is obtained with the lowest base frequency necessary to maintain the preset cell current value. The base frequency increases by decreasing the pulse amplitude, using the potentiometer "pulse voltage".
Under ideal conditions the voltage can be reduced to 15–10V and still maintain a frequency not higher than 5 KHz: this increases the linearity range and also improves the signal/noise ratio.

5. When the frequency has reached a constant value, switch the recorder on and set baseline zero using knob (3).
The system is now ready to operate.

NOTE: It is important to monitor the displayed base frequency since it indicates, under constant conditions, the operative status of the whole system GC-detector.

VII CONSTANT FREQUENCY MODE (CF)

To operate in this mode proceed as follows:

1. Press button (1) and select pulse width using button (11): 1 μs for nitrogen and 0.1 μs for argon/methane.
2. Set a voltage of 50V on the potentiometer "pulse voltage" (10). This value can be reduced to 20–15V if the system is sufficiently clean.
3. Set a pulse frequency in the range 0–50 KHz by means of knob (3) and read frequency on display (4). As preliminary condition for operating frequency set a value of 5 KHz corresponding to a pulse period of 200 μs.
4. Zero recorder baseline by means of the potentiometer (5) and read standing current value (nA) on the same potentiometer dial.
As an indication it might be considered that a 50V amplitude and 5KHz frequency should generate a current greater than 1nA. Wait until the baseline is stable and the current is constant before starting analytical operations.

NOTE: A decrease in pulse frequency increases the probability of capturing free electrons by electronegative substances and therefore also detector response, provided that the standing current remains at an acceptable level.

VIII DETECTOR MAINTENANCE

The electron capture detector ECD-40, if properly operated, is highly contamination resistant thanks to the design of its internal geometry.

However certain critical operating conditions of the column or other causes, can produce, on a long term, contamination of the collector electrode. The symptoms of this condition are an excessive increase of the base frequency in CC mode, or a decrease of the standing current in CF mode, also a baseline instability occurs when changing operating parameters.

WARNING: Any maintenance or service operations involving access to the radioactive source must be performed ONLY by authorised licensed personnel experienced in handling radioactive material. Please contact your local Carlo Erba Service Department for any operations involving cleaning or replacement of the radioactive cell.

Symptoms	Diagnosis	Remedies
1. High base frequency in CC mode or low standing current in CF mode.	a) Impure gas supply.	a) Use Research grade gases and filters to trap water and oxygen.
	b) Dirty septum.	b) Clean or replace septum.
	c) Column seals not correctly chosen.	c) Use graphite, graphitised vespel or ceramic/PTFE ferrules.
	d) Excessive column bleed.	d) Conditioning of column is needed.
	e) Carrier gas leak.	e) Check for leaks.
	f) Leak in detector assembly.	f) Check ECD tightness: turn gently the allen screws (see 32, Fig 4.1.2.5. until the base frequency returns to the normal value (CC mode).
	g) Contaminated collecting electrode.	g) Clean the electrode surface as described in VIII.
	h) Chemically contaminated radioactive source.	h) See VIII.
	i) Pulse width selector in wrong position.	i) Select 1 μ sec for nitrogen and 0.1 μsec for Ar/CH_4.
2. Negative dips after peaks.	a) Contaminated collecting electrode.	a) See VIII.
	b) Contaminated radioactive source.	b) See VIII.
3. Baseline drifts when changing voltage value.	a) Contaminated collecting electrode.	a) See VIII.
4. No base frequency is displayed in CC mode (no current is measured in CF mode).	a) Detector conditioning time too short.	a) Increase time of conditioning.
	b) ECD Control Module disconnected.	b) Check good connection.
5. Detector not heated.	a) Temperature sensor faulty.	a) Replace.
	b) Heater disconnected.	b) Check connection to the Control Module.
	c) LED alarm ON (power cut off).	c) Switch module OFF and ON.
	d) Heater faulty.	d) Replace.
	e) Temperature thumbwheel switch faulty.	e) Replace.
6. LED alarm is on.	a) Detector heater disconnected.	a) Check connection.
	b) Detector overheating (limit at temperature sensor: 430°C).	b) Contact Service Engineer.
	c) Pt 100 sensor faulty.	c) Replace.
	d) Heater filament faulty.	d) Replace.
7. Round top peaks.	a) Detector outside of linear range.	a) Operate CC mode. Decrease pulse voltage. Decrease sample concentration.
8. Negative peaks.	a) Presence of high amounts of organic compounds (co-extracts).	a) Increase reference current (above 1.8–2 nA CC mode).

X LIST OF SPARE PARTS

A list of the recommended spare parts is given in this section in order to facilitate the identification of the requested part.

To order the parts, it is always helpful to state the model and serial number of the instrument for which the part is destined.

Since our policy with reference to spares is one of continuous development, we reserve the right to substitute units or parts of alternative specification.

Fig	Ref	Description	Code Number
4.1.2.A	1	Computing-Integrator. 3 pin connector	238 16043
4.1.2.4	–	*Radioactive source ^{63}Ni	231 05001
	–	*Maintenance kit for ECD-40	190 04524
4.1.2.5	25	Jet for ECD-40 (set of 2) (standard jet)	404 02000
	–	PTFE O-ring for jet 404 02000 (set of 10)	290 30903
	–	High temperature jet for ECD with aluminium seals (above 280°C) (optional jet)	404 04401
	–	Aluminium seals for jet 404 04401	290 23609
	26	Brass nut for ECD-40	350 01119

*Any maintenance or replacement affecting the radioactive cell is prohibited unless the user is specifically licensed to handle radioactive material, alternatively, all repairs to this detector must be performed by authorised licensed CARLO ERBA STRUMENTAZIONE technical personnel.

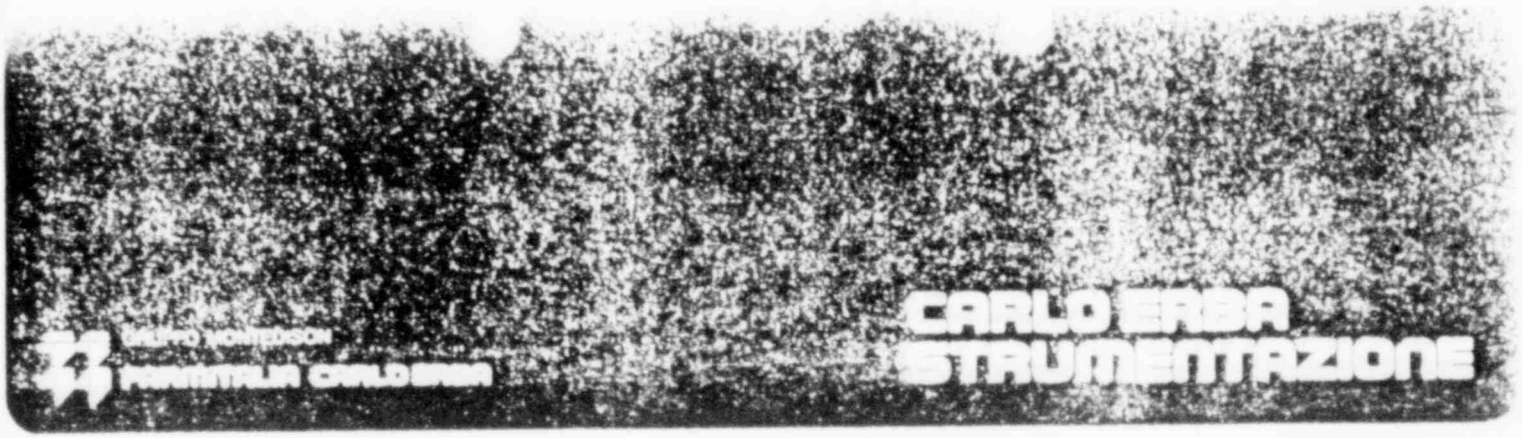
Fig	Ref	Description	Code Number
4.1.2.2	1,2	Pushbutton complete	236 47555
	1,2	Green LEDs	305 21100
	3	Frequency knob	311 00321
	3	Locking knob	283 00600
	4	Digital frequency display	236 47570
	5	Double function potentiometer knob	311 00600
	6	Attenuator knob	311 00330
	7	Detector temperature thumbwheel switches (one sector only)	236 47558
	8	Yellow LED (set of 5)	305 21102
	9	Alarm green LED (set of 5)	305 21100
	10	Multidial potentiometer knob	311 00600
	11	Green LED (set of 5)	305 21100
4.1.2.3	12	Main ON/OFF power switch	237 12322
	18	Detector coaxial cable complete	230 32030
	19	Fuses 2A rating	282 04019
	21	Main power cable	230 06400
INTERNAL COMPONENTS			
	–	Main transformer T1	413 12041
	–	Transformer T2	413 12042
	–	SSR 400	236 47560
	–	TPL1 A	236 19902
	–	MBE 400	236 47565
	–	Pulse voltage potentiometer (knob not included)	336 00319
	–	Reference current potentiometer (knob not included)	336 00319

APPENDEX "C"

ECD-40 ELECTRICAL TESTING PROCEDURE

see Figg. SC 204 and FC 369

1) Check good connection and insulation of electrodes.
2) Check good insulation toward ground.
3) Check the Pt 100 sensor.
4) Check the two serial connetted heaters (24V - 25W).
5) Check the coaxial cable and connect it to the detector.
6) Check the Pt 100 sensor at pins J and K of the connector.
7) Check the heaters at pins B and D.
8) Connect the detector to the Control Module 400 for the final test.
9) Check the correct operation of the overheating alarm system.
   (see diagrams SE 528 - 42100701 - SE 523 and SE 525 for the circuitary inspection).


CARLO ERBA
STRUMENTAZIONE

Item 7

WIPE TEST METHOD

Procedure

1. Wet cotton wool with methanol.
2. Wipe outer surface of ECD.
3. Remove exit tube from top of ECD.
4. Wipe the bottom of the exit tube with moist cotton wool.
5. Remove the 2 screws retaining the tower cap and remove.
6. Wipe the bottom cylindrical surface of the tower cap and place the swab in a marked vial.
7. Repeat steps 1-6 with a new cotton wool swab and place in a clear marked vial.
8. Reassemble the ECD and carry out pneumatic leak check as detailed.
9. Submit the marked vials containing the swabs for measurement using a liquid scintillation counter.

Notes:

a. The wipe test should be performed on the ECD at room temperature.
b. Radioactivity should be measured on a tri-varb 460C spectrometer set at 70KeV (lower level 1-upper level 70) to encompass the max. beta energy of 67KeV Ni63. The test and background should be measured for 10 minutes.
c. Suitable precautions should be taken by the individual performing the test.
d. The test should only be carried out by designated personnel.

INTERPRETATION - RESULTS

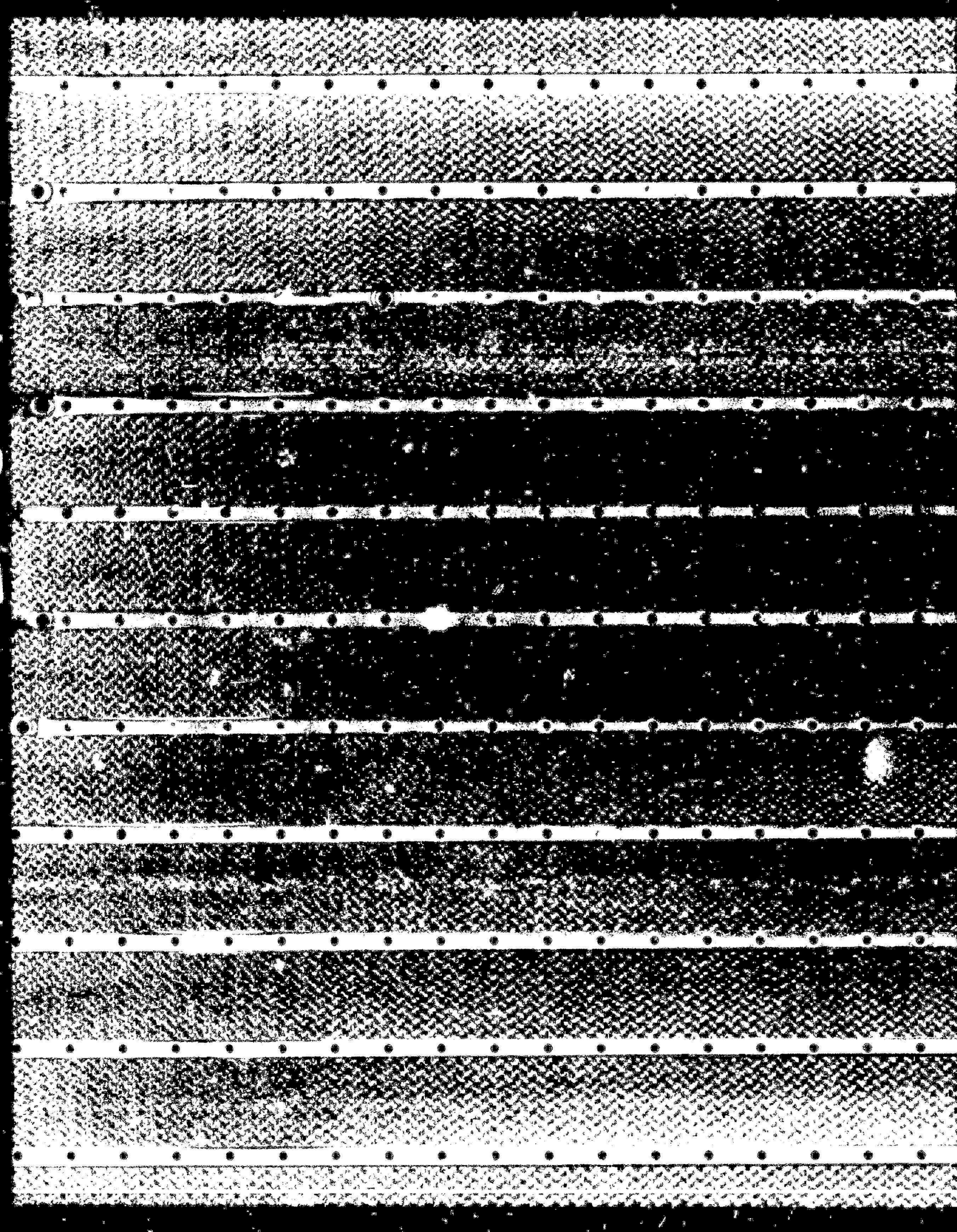
The test sample shall not exceed a baseline criterian which is the sum of the background count rate plus 20times the standard deviation of the background count rate.

Test sample $\leq$ background count rate x 20 gamma.

LIMITS

For a source containing 10mCi of Ni63 the acceptance limit is 5×10^{-3}uCi (0.18kBq)

APPENDIX "D"



ECD-40 RADIATION DOSE MEASUREMENT

The measurements of radiation dose have been carried out around the ECD installed on a gas chromatograph during typical use (Ratemeter Berthold TOL/E).

ECD-40 Serial number : 188566

- Dose rates at 5 cm around THE ECD: less than 0.03 mR/h
- Dose rate at ECD vent : less than 0.03 mR/h
- Dose rate at surface contact of ECD walls (at room temperature) : less than 0.03 mR/h

Certificate number : 305/85

Date : January 9, 1985

APPENDIX "D"

GRUPPO MONTEDISON
FARMITALIA CARLO ERBA

CARLO ERBA STRUMENTAZIONE

ECD WIPE TEST CERTIFICATE

Certificate Number: 3922/38

Wipe Test Date: 14 Dec. 1984

Source Serial Number: 2350

Radionuclide: Ni-63 10 mCi (370 MBq)

Detector Model: ECD-40

Detector Serial Number: 198318

ASSAY RESULTS*

Radionuclide	Observed Count Rate (cpm)** Gross	Background	Net	Activity (µCi)**
Ni-63 A + B	361	18	343	4.3×10^{-3}

* Method described overleaf

** Sum of measurements taken

Analysis of Results

The radioactive integrety of the Detector is within the stated limits. The detectable activity is less than 5×10^{-3} µCi.

Wipe Test Performed by: [illegible] Date: 13 Dec 1984

Analysis Performed by: [illegible] Date: 14 Dec 1984

ECD WIPE TEST

The ECD Wipe Test detailed overleaf was carried out under a controlled procedure as reported below.

A PROCEDURE

1) Wet cotton wool swab with methanol.
2) Wipe the outer surface of the ECD.
3) Remove metal exit tube from top of ECD (if using multi detector configuration remove the Jet).
4) Wipe the bottom of the exit tube (Jet).
5) Remove the 2 screws retaining the tower cap.
6) Wipe the bottom cylindrical surface and place the swab in a marked vial.
7) Repeat twice steps 1 - 6 with new cotton woll swabs and place in clean marked vials.
8) Reassemble the ECD and perform a pneumatic leak check as detailed in the ECD 40 manual.
9) Submit the marked vials containing the swabs for the radioactivity measurement using a liquid scintillation counter.

Notes: a) The Wipe Test should be performed on the ECD at room temperature.

b) Radioactivity should be measured by a TRI-CARB 460-C spectrometer (or similar) set at 70 keV (lower level 1- upper level 70) to encompass the maximun beta energy of 67 keV of ^{63}Ni. The test and background samples should be measured for 10 min.

c) Suitable precautions should be taken by the individual performing the test.

d) The test should only be carried out by authorised personnel.

B LIMITS

For a source containing 10 mCi of ^{63}Ni the acceptance limit is 5×10^{-3} uCi (0.18 KBq).

GRUPPO MONTEDISON
FARMITALIA CARLO ERBA

CARLO ERBA
STRUMENTAZIONE

ECD TEST REPORT

DETECTOR MOD. ECD 40 Serial number 198818

Nickel-63 radioactive source (10 mCi) Serial number 2350

Source contamination-wipe test : less than 0.005 nCi

Detector leak test-pneumatic : leak free

OPERATING CONDITIONS

ECD CONTROL MOD. 400

Detector Temperature : 350°C

Operational Mode : Constant Current
- Pulse Width : 1 µsec.
- Pulse Amplitude : 50V
- Reference Current : 1 nA
- Base Frequency : 2.5 KHz

Attenuation : x 64

HRGC 5300 MEGA SERIES

Capillary column : glass, 15 m length, 0.32 mm ID, OV-1 0.1 µm film thickness

Column temperature : 110°C (1 min) to 230°C at 25°C/min

Injector temperature : 250°C

Carrier gas - Helium : 2 ml/min

Auxiliary gas - Nitrogen : 30 ml/min

Injection type : Splitless
- Septum purge (T) : 3 ml/min
- Split vent (B) : 10 ml/min

Analytical checkout with 1 µl hexane solution:

1) γ -Lindane 40 pg/µl
2) Heptachlor 65 pg/µl
3) p,p' - DDE 45 pg/µl

20.12.1984

Tecno

Amersham International plc
White Lion Road Amersham
Buckinghamshire England HP7 9LL

telephone Little Chalfont (024 04) 4444
cables Activity Amersham telex
telex 83141 ACTIVA G

Radioactive source test report

product code	description	nominal activity	customer
NBCQ7086	NICKEL-63 BETA FOIL	50 10 MILLICURIE. (370 MEGABQ)	WEGMUELLER AND CO LTD POB 260 CH-8058 ZUERICH SWITZERLAND. FOR: PRODOTTI GIANNI MILAN.
AI item no. K15053			

BSI/ISO classification •	special form certificate no.	recommended working life		customer's order no.
NOT TESTED	NONE	NOT ASSESSED	903 1	R18685C

serial number	measurement check	measurement check date	leakage test	date passed	leakage test	date passed	contamination test	date passe
2094 BT		21 JUNE 84					20 SEPT 84	
2349 BT		8 OCT 84					10 OCT 84	
2350 BT		8 OCT 84					11 OCT 84	
2351 BT-2358 BT		8 OCT 84					10 OCT 84	
2362 BT		8 OCT 84					10 OCT 84	
2392 BT-2399 BT		26 JULY 84					20 SEPT 84	
2402 BT-2415 BT		6 AUG 84					20 SEPT 84	
2416 BT-2431 BT		7 AUG 84					20 SEPT 84	

notes

THESE SOURCES ARE UNSEALED, HANDLE WITH CARE. AVOID ABRADING ACTIVE FACE.

signed A. L. Stroud

date 30 OCT 84

• this classification complies with BS.5288:1976, which is in agreement with ISO.2919 (see overleaf for definition and description of tests)

Amersham

ISO Classification

The International Organization for Standardization (ISO) has proposed a system of classification of sealed radioactive sources based on safety requirements for typical uses (see ISO 2919).

"This system provides a manufacturer of sealed radioactive sources with a set of tests to evaluate the safety of his products under working conditions. It also assists a user of such sealed sources to select types which suit the application he has in mind, especially where protection against the release of radioactive material is concerned".

The tests to which prototype sources are subjected are listed in Table 1.
Each test can be applied in several degrees of severity. Test results are expressed as a five figure code to indicate the severity of the tests.
These figures are preceded by the letter C or E to show whether the source activity is less than or greater than certain limits. These limits depend upon the toxicity, solubility and reactivity of the active component of the source.
C indicates that the activity level of the source does not exceed the prescribed limit and
E that the limit is exceeded.

Table 1. Classification of sealed source performance standards

Test	Class 1	2	3	4	5	6
Temperature	No test	−40 °C (20 min) + 80 °C (1 h)	−40 °C (20 min) + 180 °C (1 h)	−40 °C (20 min) + 400 °C (1 h) and thermal shock 400 °C to 20 °C	−40 °C (20 min) + 600 °C (1 h) and thermal shock 600 °C to 20 °C	−40 °C (20min) + 800 °C (1 h) and thermal shock 800 °C to 20 °C
External pressure	No test	25kPa absolute to atmospheric pressure	25kPa absolute to 2 MPa absolute	25kPa absolute to 7MPa absolute	25kPa absolute to 70MPa absolute	25kPa absolute to 170MPa absolute
Impact	No test	50g from 1m	200g from 1m	2kg from 1m	5kg from 1m	20kg from 1m
Vibrations	No test	30min 25Hz to 500Hz at 5gn peak amplitude	30min 25Hz to 50Hz at 5gn peak amplitude and 50Hz to 90Hz at 0·635mm amplitude peak to peak and 90Hz to 500Hz at 10gn	90min 25Hz to 80Hz at 1·5mm amplitude peak to peak and 80Hz to 2000Hz at 20gn		
Puncture	No test	1g from 1m	10g from 1m	50g from 1m	300g from 1m	1kg from 1m

Notes to Table 1

1. Details of the testing procedures are given in ISO 2919 and BS 5288. A further class X can be used where a special test procedure has been adopted.
2. External pressure
   100kPa = 1 atmosphere (approx.)
3. Impact test
   The source, positioned on a steel anvil, is struck by a steel hammer of the required weight, the hammer has a flat striking surface, 25mm diam. with the edges rounded.
4. Puncture test
   The source, positioned on a hardened steel anvil, is struck by a hardened pin, 6mm long, 3mm diam. with hemispherical end, fixed to a hammer of the required weight.

Quality control

Tests for leakage and contamination

Stringent tests for leakage are an essential feature of radioactive sources production. The methods adopted depend on the design and intended application of the source, and also on statutory requirements. Where necessary, tests can be specially modified to meet particular requirements.
The standard methods used for testing radiation sources are listed below.

Wipe test A
The source is wiped with a swab or tissue, moistened with methanol or water, the activity removed is measured. Acceptance limit 0·005μCi (0·185Bq).
(This test conforms to BS 5288 App D 2 1).

Wipe test B
The source is wiped with a swab or tissue, moistened with methanol or water, the activity removed is measured. Acceptance limit 0·05μCi (1·85Bq).

Bubble test D
The source is immersed in water or a suitable liquid and the pressure reduced to 100mm of mercury (13kPa).
No bubbles must be observed.
(This test conforms to BS 5288 App D 3 1 and it has been demonstrated that the test is suitable for sources with free volumes greater than 50mm³ (Amersham International Technical Report No. 8)).

Immersion test E
The source is immersed in water or other suitable liquid at 50 °C for 4 hours and the activity in the liquid measured. Acceptance limit 0·005μCi (0·185Bq).
(This test conforms to BS 5288 App D 2 3).

Immersion test M
The source is immersed in water which is raised to 100 °C and held at that temperature for 10 min. The water is then removed, the source cooled and rinsed using fresh water. These operations are repeated twice, boiling in the water from the previous rinsing operation. If the activity detected in all the liquid collected is less than 0·005μCi (0·185Bq) the source is considered to be leak free.
(The test conforms to BS 5288/App. D 2 4).

Helium leak test H
A mass spectrometer is used to detect helium leakage from sources helium filled prior to welding.
Acceptance limit 10^{-7} mbar l s^{-1}
(This test exceeds the requirement of BS 5288/App. D 3 4).

Helium pressurisation test S
A mass spectrometer is used to detect helium leakage from sources previously pressurised in helium after encapsulation. Acceptance limit 10^{-6} mbar l s^{-1} for non-leachable solid inserts and 10^{-7} mbar l s^{-1} for all others.
(This test exceeds the requirement of BS 5288/App. D 3 5).

Radon emanation test K
The source is immersed in a solution of phosphor in an organic liquid under vacuum, the leakage of radon is measured by liquid scintillation counting.
(DWJ/HI/D.J. Radiochemical Centre Report R176).
The acceptance limit corresponds to about 5x10^{-6} Ci per 24 hours (2Bq per 24 hours).
This test exceeds the requirement of BS 5288/App. D 2 6).

Krypton emanation test P
The source is held under reduced pressure for 24 hours. The contents of the chamber is analysed for Krypton by scintillation counting. The test is repeated after at least 7 days. Acceptance limit 0·1μCi (3·7kBq).

Performance requirements for typical uses

Typical applications in which sealed radioactive sources may be used, with minimum performance requirements are also given in ISO 2919 (see Table 2 below). These recommendations take into account normal usage and reasonable accidental risks, but do not include exposure to the risk of fire, explosion or corrosion.

Table 2. Sealed source performance requirements for typical uses

Sealed source use		Sealed source test and class Temperature	Pressure	Impact	Vibration	Puncture
Industrial radiography	Unprotected source	4	3	5	1	5
	Source in device	4	3	3	1	3
Gamma gauges (medium and high energy)	Unprotected source	4	3	3	3	3
	Source in device	4	3	2	3	2
Beta gauges and sources for low energy gamma gauges or X-ray fluorescence analysis (excluding gas filled sources)		3	3	2	2	2
Oil well logging		5	6	5	2	2
Portable moisture and density gauges (including hand held or daily transported)		4	3	3	3	3
General neutron source application (excluding reactor start-up)		4	3	3	2	3
Calibration sources, activity greater than 30μCi (1·11MBq)		2	2	2	1	2
Gamma irradiation sources	Unprotected source	4	3	4	2	4
	Source in device	4	3	3	2	3
Ion generators (source-device combination may be tested)	Chromatography	3	2	2	1	1
	Static eliminators	2	2	2	2	2
	Smoke detectors	3	2	2	2	2
Medical	Radiography	3	2	3	1	2
	Gamma teletherapy	5	3	5	2	4
	Beta teletherapy	5	3	3	2	2
	Interstitial and intracavitary appliances*	5	3	2	1	1
	Surface applicators	4	3	3	1	2

*Sources of this nature may be subject to severe deformation in use. Manufacturers and users may wish to formulate additional or special test procedures.

If the sealed source has a 'C' classification, Table 2 can be used directly to assess the suitability of the source for the proposed application provided that there is no significant fire, explosion or corrosion hazard.

If such a hazard does exist, the user and the manufacturer have to consider the following factors to determine whether additional testing is required:
1. consequences of loss of activity,
2. quantity of active material contained in the source,
3. radiotoxicity,
4. chemical and physical form of the material and the geometrical shape,
5. environment in which it is to be used,
6. protection afforded to the source or source-device combination.

If the sealed source has an 'E' classification, Table 2 cannot be used directly.

To determine whether any additional testing is necessary, an evaluation of the fire, explosion and corrosion hazards must be made and a suitable evaluation of the use and design of the source.

Some of our source designs exceed the recommendations of Table 2 and may therefore be acceptable for the applications listed despite the 'E' classification.

Acknowledgement: Tables from BS 5288: 1976 are reproduced by permission of BSI, 2 Park Street, London W1A 2BS from whom complete copies can be obtained.

Laboratory applications

The ISO classification system does not refer explicitly to sources designed for research laboratory usage because of the wide variety of applications and environments in which such sources might be used.

Special applications

No test programme can cover all possible combinations of environments to which a source may be exposed.

Users should therefore consult our technical staff before using sources in potentially adverse environments.

IAEA Special Form

'Special Form' is a test specification for sealed sources given in the IAEA transport regulations.
(IAEA Safety Series No. 6, 1967/1973 revised edition†).

It is used in determining the maximum acceptable activities for various types of transport containers.

The required tests are:
impact test
percussion test
bending test (only for long, slender sources)
heat test.
After each test the source must be subjected to immersion testing.

The certificate (SFC) numbers given are those issued by the Dept. of the Environment, the competent authority in the UK for administering the IAEA regulations.

†1973 regulations not yet universally adopted.

Source working life

The *'recommended working life'* is our recommendation of the period within which the source should be replaced. The period given has been assessed on the basis of such factors as: toxicity of nuclide, total initial activity, source construction (eg capsule design, source insert type, etc.), half-life of nuclide, typical application environments, operational experience, test performance data, etc.

Adverse environments could affect the appearance and integrity of a source. It is the user's responsibility to regularly inspect and test the source in order to assess at what point during the 'recommended working life' the source should be replaced.

11-17 198x

CARLO ERBA
STRUMENTAZIONE

GRUPPO MONTEDISON
CARLO ERBA

REGISTRO CARICO E SCARICO SOSTANZE RADIOATTIVE

LABORATORIO RIVELATORI RADIOATTIVI

DATA DI ARRIVO	SOSTANZA RADIOATTIVA	MATRICOLA SORGENTE	ATTIVITÀ TOTALE	UTILIZZAZIONE				ELIMINAZIONE
				RIVELATORE		MANUTENZIONE	SCARTO	
				DATA	MATRICOLA			
05-11-84	Ni^{63}	2349	10 mCi	19-12-84	198807			
05-11-84	Ni^{63}	2350	10 mCi	20-12-84	198818			
05-11-84	Ni^{63}	2351	10 mCi	20-12-84	198825			
05-11-84	Ni^{63}	2352	10 mCi	20-12-84	198815			

The Radiochemical Centre Amersham

Radiation Sources Department 18 December 1979

Dear Mr Verga

Nickel-63 foils for EC detectors

As promised in my letter of 3 December, we have tested an NBC3 source in the sample detector cell at elevated temperature.

We subjected the foil to a temperature of 450°C in nitrogen for 3 days and found no measurable decrease in standing current. The springs appear to have survived the test without loss of tension.

OK

If you would like to carry out the test for a longer period we will gladly send the foil and cell to you.

Yours sincerely

D J Dwight
Electroplated and Reference Sources

Mr Verga
Carlo Erba
Via Imbonati
24 Milano
Italy

WARNING LABEL AS ON ECD 40

SHIPPING WARNING LABEL IATA

Erba Instruments Inc.

MANUFACTURING LICENSE AS GRANTED TO CARLO ERBA STRUMENTAZIONE BY ITALIAN NUCLEAR AUTHORITY.

item 1) Original license granted to Carlo Erba on 5 March, 1971 valid for 5-years.

item 2) Ammendment license granted to Carlo Erba Strumentazione on 28 December, 1974 for 5-years.

item 3) Indefinite license granted to Carlo Erba Strumentazione on 16 October, 1980 for an indefinate period.

Ministero dell'Industria
del Commercio dell'Artigianato
D.G. Fonti Energia e Ind. Base
Servizio Energia Nucleare
Divisione VI

Prot. N. 8[illegible] 15/C Allegati 1

ITEM !

Alla CARLO ERBA STRUMENTAZIONI
S.p.A.
Strada Rivoltana
20090 RODANO (Milano)

Risposta al Foglio N.°
del

OGGETTO : Carlo Erba Strumentazioni S.p.a. - Decreto interministeriale di nulla osta al commercio di cat. B (art. 34 D.P.R. 13.2.1964 n. 185).

e, p.c.: Al MINISTERO DELL'INTERNO
- D.G. Servizi Antincendi e Protezione Civile
- D.G. Pubblica Sicurezza
ROMA

Al MINISTERO DEL LAVORO E DELLA PREVIDENZA SOCIALE
D.G. Rapporti di Lavoro
ROMA

Al MINISTERO DELLA SANITA'
D.G. Servizio Igiene Pubblica
ROMA

Al PREFETTO di
MILANO

All'ISPETTORATO PROVINCIALE DEL LAVORO di MILANO

All'UFFICIO DEL MEDICO PROVINCIALE di
MILANO

All'UFFICIO PROVINCIALE INDUSTRIA COMMERCIO E ARTIGIANATO di
MILANO

Al COMITATO NAZIONALE PER L'ENERGIA NUCLEARE
Viale Regina Margherita, 125
ROMA

Con riferimento alla domanda sopraindicata si trasmette, in allegato, copia del decreto in data 16.10.1980 concernente l'autorizzazione di cui all'oggetto.

./.

IL DIRETTORE DI SEZIONE

Il Ministro dell'Industria del Commercio e dell'Artigianato

di concerto con

IL MINISTRO DELL'INTERNO

IL MINISTRO DEL LAVORO E DELLA PREVIDENZA SOCIALE

IL MINISTRO DELLA SANITA'

VISTA la legge 14 ottobre 1957 n. 1203 concernente la ratifica e l'esecuzione del Trattato istitutivo della Comunità Europea dell'Energia Atomica;

VISTA la legge 11 agosto 1960 n. 933 concernente la istituzione del Comitato Nazionale per l'Energia Nucleare, ristrutturato con legge 15.12.1971 n. 1240;

VISTA la legge 31 dicembre 1962 n. 1860 concernente l'impiego pacifico dell'energia nucleare, modificata e integrata dal D.P.R. 30.12.1965 n. 1704, dalla legge 19.12.1969 n. 1008 e dal D.P.R. 10.5.1975 n. 519;

VISTO il D.P.R. 13 febbraio 1964 n. 185 concernente la sicurezza degli impianti e la protezione sanitaria dei lavoratori e delle popolazioni contro i pericoli delle radiazioni ionizzanti derivanti dall'impiego pacifico dell'energia nucleare;

VISTI i decreti applicativi della citata legge 31.12.1962 n. 1860 e successive modifiche e integrazioni, nonchè del citato D.P.R. 13.2.1964 n. 185;

VISTA l'istanza in data 11.7.1979 e successiva documentazione integrativa con la quale la Carlo Erba Strumentazioni S.p.A. con sede legale in Rodano (Mi) - Strada Rivoltana - ha chiesto la proroga del nulla osta di categoria B, rilasciato con decreto interministeriale 28.12.1974, nonchè la variazione della sede legale da Trezzano sul Naviglio a Rodano - Strada Rivoltana;

VISTO il parere favorevole espresso dal Ministero della Sanità con nota 400.5/X-49/711 in data 29.12.1979;

VISTO il parere favorevole espresso dal CNEN con lettera prot. 31924/18 del 6.12.1979;

VISTO il parere favorevole espresso dal Ministero del Lavoro con nota 7RL21427RD dell'11.3.1980;

VISTO il parere favorevole espresso dal Ministero dell'Interno con nota 3584/85285 del 15.2.1980;

BU/pa

./.

Il Ministro
dell'Industria del Commercio e dell'Artigianato

D E C R E T A :

Art. 1

Il nulla osta all'esercizio del commercio di categoria B di sostanze radioattive rilasciato alla Carlo Erba Strumentazioni S.p.A. con decreto interministeriale 28.12.1974 è prorogato "sine die".

Art. 2

Il decreto interministeriale 28.12.1974 è da intendersi rilasciato per la nuova sede legale della Società trasferita da Trezzano sul Naviglio a Rodano - Strada Rivoltana.

Art. 3

Restano fermi - a carico della Carlo Erba Strumentazioni - tutti gli obblighi di cui al decreto 28.12.1974.

Roma 16 OTT. 1980

IL MINISTRO DELL'INTERNO

IL MINISTRO DELL'INDUSTRIA, DEL COMMERCIO E DELL'ARTIGIANATO

IL MINISTRO DEL LAVORO E DELLA PREVIDENZA SOCIALE

IL MINISTRO DELLA SANITA'

PER COPIA CONFORME

ITEM 2

MOD 25 P

MODULARIO IND. COMM. 42

Roma, 28 DIC. 1974 19

Ministero dell'Industria
del Commercio e dell'Artigianato
D.G.Forti Energia e Ind.Base
Serv.Energia Nucleare
Div.VI

Prot. N. 29817/15BIS Allegati 1

Alla Carlo IRBA S.P.A.
via L. [illegible]
SEGRATE SUL NAVIGLIO (Milano)

Risposta al Foglio N.
del 21.3.1974

OGGETTO Decreto di null. osta all'esercizio del commercio di categoria B di materie radioattive (art.34 D.P.R. 13.2.1964 n.185).

e,p.c.:

AL MINISTERO DELL'INTERNO
D.G. SERVIZI ANTINCENDI E
PROTEZIONE CIVILE ROMA

AL MINISTERO DEL LAVORO E
DELLA PREVIDENZA SOCIALE
D.G. RAPPORTI DI LAVORO ROMA

AL MINISTERO DELLA SANITA'
D.G. IGIENE PUBBLICA ROMA

AL PREFETTO DI MILANO

ALL'UFFICIO DEL MEDICO
PROVINCIALE DI MILANO

ALL'ISPETTORATO PROVINCIALE
DEL LAVORO DI MILANO

ALL'UFFICIO PROVINCIALE
INDUSTRIA COMMERCIO E
ARTIGIANATO DI MILANO

AL COMITATO NAZIONALE PER
L'ENERGIA NUCLEARE
viale Regina Margherita 125 ROMA

ALLA CARLO IRBA S.P.A.
via C. Imbonati n.24 MILANO

In riferimento alla istanza sopraindicata, si trasmette copia del decreto in data 28.12.1974, con il quale a codesta Società è stato trasferito il nulla osta di cui all'oggetto, già rilasciato alla Carlo IRBA S.p.a. con sede in Milano, via C. Imbonati n.24, con il decreto in data 2 marzo 1971.

IL DIRETTORE DELLA DIVISIONE

Il Ministro Segretario di Stato

PER L'INDUSTRIA, PER IL COMMERCIO E PER L'ARTIGIANATO

di concerto con

IL MINISTRO PER L'INTERNO
IL MINISTRO PER IL LAVORO E PER LA PREVIDENZA SOCIALE
IL MINISTRO PER LA SANITA'

VISTA la legge 14 ottobre 1957, n.1203, concernente la ratifica e l'esecuzione del Trattato istitutivo della Comunità Europea dell'Energia Atomica;

VISTA la legge 11 agosto 1960, n.933, concernente la istituzione del Comitato Nazionale per l'Energia Nucleare;

VISTA la legge 15 dicembre 1971, n.1240, concernente la ristrutturazione del Comitato Nazionale per l'Energia Nucleare;

VISTA la legge 31 dicembre 1962, n.1860, concernente lo impiego pacifico dell'energia nucleare;

VISTO il D.P.R. 13 febbraio 1964, n.185, concernente la sicurezza degli impianti e la protezione sanitaria dei lavoratori e delle popolazioni contro i pericoli delle radiazioni ionizzanti derivanti dall'impiego pacifico dell'energia nucleare;

VISTO il proprio Decreto 13 novembre 1964, (G.U. n.297 del 1° dicembre 1964) concernente l'approvazione del modello dello speciale registro previsto dall'art.36 del citato D.P.R. n.185;

VISTO il D.P.R. 30 dicembre 1965, n.1704, concernente modifiche ed integrazioni alla citata legge 31 dicembre 1962, n.1860;

VISTO il D.M. 15 giugno 1966 (G.U. n.219 del 3 settembre 1966), concernente la determinazione delle attività delle sostanze radioattive ai fini della classificazione degli esercizi commerciali, di cui all'art.32 del D.P.R. 13.2.1964, n.185;

VISTO il D.M. 26 ottobre 1966 (G.U. n.302 del 30 novembre 1966), concernente le norme relative alla procedura di rilascio del nulla osta prescritto dall'art.34 del citato D.P.R. n.185, per gli esercizi di categoria 2 autorizzati al commercio dei minerali, delle materie grezze e delle materie radioattive, ai sensi dell'art.4 della legge 31.12.1962, n.1860;

./.

VISTO il D.M. 6 giugno 1968 (G.U. n.220 del 30 agosto 1968), concernente la determinazione delle dosi e delle concentrazioni massime ammissibili ai fini della protezione sanitaria dei lavoratori dalle radiazioni ionizzanti;

VISTO il D.P.R.5 dicembre 1969 n.1303, concernente la determinazione delle quantità di radioattività, delle attività specifiche o concentrazioni e delle intensità di dose di esposizione soggette alle prescrizioni del citato D.P.R. 13 febbraio 1964, n.185;

VISTO il D.M. 2 febbraio 1971 (G.U. n.58 del 6 marzo 1971), concernente la determinazione dei valori delle dosi massime ammissibili e delle concentrazioni massime ammissibili, nonchè dei valori dell'efficacia biologica relativa per la popolazione nel suo insieme e per i gruppi particolari della popolazione, ai fini della protezione contro i pericoli derivanti dalle radiazioni ionizzanti;

VISTO il D.P.R. 12 dicembre 1972 n.1150, concernente la determinazione delle modalità per l'iscrizione negli elenchi degli esperti qualificati e dei medici autorizzati incaricati della sorveglianza fisica e medica della protezione dalle radiazioni ionizzanti;

VISTO il proprio decreto in data 2 marzo 1971 con il quale alla Carlo Erba S.p.A. con sede in Milano, via Carlo Imbonati n. 24 é stato rilasciato il nulla osta prescritto dall'art. 34 del citato D.P.R. 13.2.1964 n. 185 per l'esercizio del commercio di categoria B di materie radioattive presso i locali del Laboratorio "Divisione Apparecchi Scientifici" ubicato in Rodano (Milano);

VISTA la domanda in data 21 marzo 1974 con la quale la detta Società - a seguito di atto di concentrazione 28-/2/1974 rogito notaio Bruni - rep. 20033/3891 con cui il predetto Laboratorio "Divisione Apparecchi Scientifici" ubicato in Rodano (Milano) é stato incorporato fra le attività della società consociata Carlo Erba Strumentazioni S.p.A. con sede in Trezzano sul Naviglio (Milano) via Leonardo da Vinci n. 149 - ha chiesto che il menzionato decreto 2 marzo 1971 sia intestato a questa ultima Società;

VISTA la domanda in data 21.3.1974 con la quale la predetta Carlo Erba Strumentazioni S.p.A. ha chiesto l'intestazione a proprio nome del decreto 2.3.1971, già rilasciato alla Carlo Erba S.p.A.;

VISTO il proprio decreto in data 20 aprile 1974, con il quale la Carlo Erba Strumentazioni S.p.A. é stata autorizzata al commercio di materie radioattive, ai sensi dell'art. 4 della legge 31.12.1962 n. 1860;

./.

VISTO il sopracitato atto di concentrazione 28.2.1974, rogito notaio Bruni, rep. 20033/3891;

D E C R E T A :

Art. 1

Il nulla osta all'esercizio del commercio di categoria B di materie radioattive, già rilasciato alla Carlo Erba S.p.A. con sede in Milano, via Carlo Imbonati n. 24 con decreto in data 2.3.1971, ai sensi dall'art. 34 del D.P.R. 13.2.1964 n. 185, é trasferito alla Carlo Erba Strumentazioni S.p.A., con sede in Trezzano sul Naviglio (Milano), via Leonardo da Vinci n. 149 e Stabilimento in Rodano (Milano).

Art. 2

Fermi restando - a carico della Carlo Erba Strumentazioni S.p.A. - gli obblighi derivanti dagli articoli 2 e 3 dello anzidetto D.M. 2.3.1971, il nulla osta di cui al precedente art. 1 é valido per un periodo di cinque anni dalla data del presente decreto e può essere prorogato.

Roma, 28 dicembre 1974

IL MINISTRO
PER L'INTERNO

IL MINISTRO PER L'INDUSTRIA, PER IL COMMERCIO E PER L'ARTIGIANATO

IL MINISTRO PER IL LAVORO
E LA PREVIDENZA SOCIALE

IL MINISTRO
PER LA SANITA'

ITEM 3

MODULARIO I. C. 22

MOD. 25 P

Roma, 5 MAR. 1971 19

Ministero dell'Industria del Commercio e dell'Artigianato
D.G.F.E.I.B.
Servizio V-Divisione XI

Alla CARLO ERBA S.p.A.
Via Carlo Imbonati,24
MILANO

Prot. N. 2U0546 | 15 C Allegati 1

Risposta al Foglio N.
del

OGGETTO: Carlo Erba S.p.A. - Milano - Nulla osta all'esercizi del commercio di materie radioattive-Cat.B(Art.34 del D.P.R. 13/2/1964,n.185).

In esito all'istanza dell'11/2/1970 concernente l'oggetto, si comunica che con decreto ministeriale del 2 marzo 197 che si allega in copia, a codesta Società è stato concesso il nulla osta all'esercizio del commercio di materie radioattive da svolgere presso il proprio stabilimento, sito in Rodano (Milano).

Si richiama l'attenzione di codesta Società sull'osservanza degli obblighi richiamati negli artt.2 e 3 del citato decreto ministeriale.

IL DIRETTORE DI DIVISIONE
F. Alessandro

D'ON/ac

.5.12
01588

Il Ministro Segretario di Stato

PER L'INDUSTRIA, PER IL COMMERCIO E PER L'ARTIGIANATO

di concerto con

IL MINISTRO PER L'INTERNO

IL MINISTRO PER IL LAVORO E PER LA PREVIDENZA SOCIALE

IL MINISTRO PER LA SANITA'

VISTA la legge 14 ottobre 1957, n. 1203, concernente la ratifica e l'esecuzione del Trattato istitutivo della Comunità Europea dell'Energia Atomica;

VISTA la legge 11 agosto 1960, n. 933, istitutiva del Comitato nazionale per l'energia nucleare;

VISTA la legge 31 dicembre 1962, n. 1860, sull'impiego pacifico della energia nucleare;

VISTO il D.P.R. 13 febbraio 1964, n. 185, sulla sicurezza degli impianti e sulla protezione sanitaria dei lavoratori e delle popolazioni contro i pericoli delle radiazioni ionizzanti derivanti dall'impiego pacifico dell'energia nucleare;

VISTO il D.P.R. 30 dicembre 1965, n. 1704, recante modifiche e integrazioni alla legge 31 dicembre 1962, n. 1860;

VISTO il D.M. 15 giugno 1966, con il quale sono state determinate le quantità totali di radioattività ai fini della classificazione degli esercizi commerciali, ai sensi dell'art. 32 del D.P.R. 13 febbraio 1964, n. 185;

VISTO il D.M. 26 ottobre 1966, con il quale sono state emanate le norme di procedura per il rilascio del nulla osta prescritto dall'art. 34 del sopracitato D.P.R. n. 185, per gli esercizi commerciali di categoria B autorizzati al commercio dei minerali, delle materie grezze e delle materie radioattive, ai sensi dell'art. 4 della legge 31.12.1962, n. 1860;

VISTO il D.M. 9 dicembre 1968, con il quale alla Società Carlo Erba S.p.A. é stato concesso il nulla osta, prescritto dall'art. 34 del D.P.R. 13 febbraio 1964 n. 185 per l'esercizio del commercio - categoria B - delle materie grezze e radioattive da svolgere presso i laboratori "Divisione Apparecchi Scientifici" e "Divisione Chimica Industriale" ubicati in Milano, via Carlo Imbonati, n. 24;

VISTA la domanda, datata 11.2.1970, con la quale la predetta Società, premesso di aver trasferito l'esercizio del commercio di materie radioattive dai

./.

D'ON/ja

locali siti in Milano, via Carlo Imbonati, n. 24, ai locali ubicati presso il proprio stabilimento di Rodano (Milano), ha chiesto il nulla osta ex art. 34 del citato D.P.R. n. 185 relativo a questi ultimi locali;

VISTA la nota n. 9759/18 Y 3 in data 27.7.1970 con la quale il Comitato nazionale per l'energia nucleare ha espresso parere favorevole in merito al rilascio della suddetta autorizzazione;

VISTA la lettera in data 25.9.1970, con la quale la Carlo ERBA S.p.A. ha fornito precisazioni in relazione alla richiesta di assicurazioni avanzata con nota del Ministero dell'Industria, del Commercio e dell'Artigianato n. 741396/15-C dell'8.9.1970;

D E C R E T A :

Art. 1

Alla Carlo ERBA S.p.A. con sede in Milano, via Carlo Imbonati n. 24, è concesso il nulla osta, prescritto dall'art. 34 del D.P.R. 13 febbraio 1964, n. 185, sull'idoneità dell'ubicazione, dei locali di esercizio, delle attrezzature e della qualificazione del personale addetto, per l'esercizio del commercio - Categoria B - di materie radioattive, precisate nella domanda della citata Società e nella relativa documentazione, richiamata nelle premesse, da svolgere presso i locali ubicati nel proprio stabilimento, sito in Rodano (Milano).

Art. 2

La concessione del nulla osta rimane subordinata alla osservanza delle norme sull'impiego pacifico dell'energia nucleare (Legge 31.12.1960, n. 1860; D.P.R. 30.12.1965, n. 1704) e sulla sicurezza degli impianti e sulla protezione sanitaria dei lavoratori e delle popolazioni contro i pericoli delle radiazioni ionizzanti derivanti dall'impiego pacifico dell'energia nucleare (D.P.R. 13.2.1964,n.185) e, in particolare, alle condizioni:

1) - che sia garantita l'osservanza delle dosi e delle concentrazioni massime ammissibili stabilite dal D.M. 6 giugno 1968 (G.U. n. 220 del 30.8.1968), adottato in esecuzione dell'art. 87 del citato D.P.R. n. 185;

2) - sia garantito il rispetto delle norme interne di sicurezza e di protezione sanitaria predisposto dalla ditta medesima ai sensi dell'art. 61 (punto b) del D.P.R. 13.2.1964, n. 185;

3) - sia assicurata la disponibilità, nel locale di manipolazione delle sorgenti radioattive, di idonea strumentazione dosimetrica di fisica sanitaria, da mantenere in condizioni di continua efficienza e da tarare periodicamente, con frequenza non superiore all'anno;

4) - siano comunicati all'Ispettorato Medico Centrale del Lavoro ed all'Ispettorato provinciale del lavoro di Milano i nominativi e le qualifiche professionali delle persone che attendono - in attesa dell'emanazione dei decreti di applicazione degli artt. 71 e 76 del D.P.R. 13.2.1964, n. 185, ai compiti della sorveglianza medica dei lavoratori.

./.

Art. 6)

E' fatto obbligo alla CARLO ERBA S.p.A. di dare tempestiva comunicazione al Ministero dell'industria, del commercio e dell'artigianato delle eventuali variazioni dello stato di fatto e di diritto, precisato negli atti prodotti dalla Società stessa per ottenere il presente nulla osta, che comportino sostanziali modifiche all'esercizio del commercio delle materie grezze e delle materie radioattive.

Roma, li 2/3/71

IL MINISTRO
PER L'INDUSTRIA, PER IL COMMERCIO
E PER L'ARTIGIANATO

IL MINISTRO PER L'INTERNO

IL MINISTRO
PER IL LAVORO E PER LA PREVIDENZA
SOCIALE

IL MINISTRO PER LA SANITA'

V. P. P.

BETWEEN: William O. Mi..er, Chief
License Fee Management Branch
Office of Administration

John E. Glenn, Chief
Nuclear Materials Section B
Division of Engineering and
Technical Programs

LICENSE FEE TRANSMITTAL

A. REGION I More Money Needed

1. APPLICATION ATTACHED

Applicant/Licensee: Erba Instruments, Inc

Application Dated: 2/19/85

Control No.: 03450

License No.: New

2. FEE ATTACHED

Amount: $210.00 + 20 rec'd 3/13/85

Check No.: 661

3. COMMENTS

device eval. of use possession + dist.

~~Manufacturing~~ & distribution

Signed Brenda Platchek

Date 2/25/85

B. LICENSE FEE MANAGEMENT BRANCH

1. Fee Category and Amount: 9A($1,600) 3N($930) 3P($230)

2. Correct Fee Paid. Application may be processed for:

Amendment _____

Renewal _____

License ✓

Signed Frances Brown

Date 4/17/85

4/19/85

REGION I FORM 213
(MARCH 1983)

"SECTION COPY"

Service $930
Device 1600
Possession 230

2760
210
2550

BANK OF BOSTON
THE FIRST NATIONAL BANK OF BOSTON

5-39
110

Erba Instruments Inc.
4 Doulton Place
Peabody, MA 01960
Telephone (617) 535-5986
TWX 710-3470122

DATE	CHECK NO.	CHECK AMOUNT
2/19/85	661	$240.00

PAY TO THE ORDER OF ▶ U. S. Nuclear Regulatory Commission - Region 1

AUTHORIZED SIGNATURE

⑆011000390⑆ 518⑈ 70306⑈

REMITTANCE ADVICE

DETACH BEFORE DEPOSITING

ERBA INSTRUMENTS INC.

VENDOR NO

VENDOR NAME

TRANSACTION DATE	REFERENCE	GROSS AMOUNT	DEDUCTION	NET AMOUNT

CHECK DATE	CHECK NO.	TOTAL GROSS	TOTAL DEDUCTION	CHECK AMOUNT