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GAS-FLOW MEASURING HEAD FOR ELECTRON SPIN RESONANCE SPEKTROMETER

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ABSTRACT

The main units of the simple gas flow measuring head applied to an ESR spectrometer are the evaporator connected to the helium container, the miniature quartz heat isolating tube, the sample holder, the electronic temperature regulator and the toroidal transformer which is used to regulate the heating current of the evaporator coil.
The heating coil warms the cold gas obtained by evaporating the liquid helium to the desired temperature. The quarz heat isolating tube is connected to the metallic vacuum system built of stainless steel tubes by para-rubber tube pieces. The measuring head is constructed for rapid change of the samples at low temperature.
The main technical data are the following: sample temperature range 10-300 K, consumption of liquid helium at lowest temperatures: 1.5 l/h, temperature stability ±0.05 K.

АННОТАЦИЯ

К спектрометру ЭПР разработана простая газопроточная головка, имеющая следующие главные части: испаритель присоединенный к резервуару жидкого гелия, миниатюрная кварцевая трубка, находящаяся вблизи образца, держатель образца, электронный регулятор температуры и трансформатор типа тороида для регулировки тока испарительной катушки.
Полученный испарением жидкого гелия холодный газ нагревается до нужной температуры с помощью находящейся в испарителе нагревательной катушки. Кварцевая термоизолирующая трубка паракаучуковыми звеньями соединена с блоками, имеющими кожух термоизоляционного вакуума и построенными из тонкостенных изготовленных из нержавеющей стали трубок.
Главные технические параметры: область регулировки температуры образца 10÷300 K, расход жидкого гелия при минимальной температуре 1,5 л/ч, стабильность температуры ±0,05 K.

KIVONAT

Az ESR spektrométerhez kidolgozott igen egyszerü gázáramos mérőfej fő egységei a hélium szállitóedényhez csatlakozó elpárologtató, a minta környezetében lévő miniatür kvarc hőszigetelő cső, a mintatartó, az elektronikus hőmérsékletszabályozó és egy toroid transzformátor a párologtató tekercs fütőáramának szabályozására.
A folyékony hélium párologtatásával nyert hideg gázt az elpárologtatóban lévő fütőtekercs a kivánt mérési hőmérsékletre melegiti. A kvarc hőszigetelő cső paragumi csődarabokkal csatlakozik a vékonyfalu rozsdamentes acél csövekből összeállitott vákuumköpenyü fém egységhez. A mintatartó egység a gyors mintacserét és a hélium gáz elvezetését biztositja.
Főbb müszaki jellemzők: a mintahőmérséklet szabályozási tartománya 10...300 K, hütőközeg fogyasztás a legkisebb hőmérsékletnél 1.5 l/h, hőmérsékletstabilitás ± 0.05 K.

The main units of the simple gas-flow measuring head applied to an electron spin resonance spectrometer are the evaporator connected to the helium container, the miniature quarz heat isolating tube, the sample holder, the electronic temperature regulator and the toroid transformer which regulates the heating current of the evaporator coil.

An SZTG-10 type /made in the USSR/ helium container is used for the apparatus. The threaded stub 1 at the end of the longer vertical section of the stainless steel evaporator is a few centimeters above the level of the liquid. A copper tube 2 closed on the bottom and having holes on the part above the helium level is screwed onto the threaded stub.

The holder of the textilbakelite evaporator coil 3 is fixed on the threaded shaft on the lower part of the copper tube. The section of the evaporator tube which is not heat isolated 4 goes into the closed copper tube. The electronic resistance of the evaporator coil is 250 Ohm, it is made of 0.15 mm diameter kanthal wire. The opening of the outer excentric tube 5 was filled with parafine at the outlet of the evaporator coil, at the outer surface of the excentric tube the gasket of the helium container is used. At the first part of the horizontal section of the evaporator the inner tube has a larger diameter and it contains the gas heating coil. The heater is made of THERMOCOAX heating wire coiled in a spiral form, its electric resistance is 150 Ohm. The gold-chromel thermocontact 10 is placed at the end of the gas heater coil. The hermetic passing 11 of the thermopair and the leads of the gasheater is assured by glueing, the demounting of the whole unit is made easy by a threaded connection 12. The heat isolating space of the evaporator is put under vacuum and closed hermetically at the opening at the other end of the horizontal section 13. The static vacuum is helped to be maintained for a long period by the active carbon 14 placed at the cold end of the evaporator. The shorter vertical section of the evaporator is made of tubes of lesser diameter and is used to connect the miniature heat isolating tube 15.

The heat isolating dewar unit 15 placed in the microwave field is made of high purity quartz tubes, the diameter of the outer tube is 10 mm, with 1 mm wall-thickness, the diameter of the inner tube is 5.5 mm with 0.5 mm wall thickness, the length of the heat isolating unit is 150 mm. The heat isolation is

assured by pumping on the space between the tubes. The remaining pressure at room temperature is less than $1x10^{-5}$ torr. The middle section of the quartz heat isolating dewar unit is placed in the TE_{102} rectangular resonator of the ESR spectrometer. The hermetic connection of the heat isolating unit to the evaporator is assured by thick para-rubber tube pieces 16.

The inner vertical tube of the double walled sample holder unit 17 heat isolated by a vacuum coating leads the sample holder, one end of its horizontal section assures connection to the helium pipeline, the other the pumping of the heat isolating vacuum space. The section of the sample holder in the microwave field is a teflon prism of triangular cross section 18, the lower part of it is a 1,5 mm diameter, 0.2 mm thick stainless steel tube 19. The temperature is measured at the sample holder by a gold-chromel thermopair 20 and a carbon resistor fixed in the way of the gas-flow outside the megnetic field. The sample 21 is placed in a circular cavity at the higher end of the triangular crossection teflon prism, the sample is in the middle line of the magnetic field. The sample may be changed in a short time by opening the screw 22.

The gas-flow measuring head is operated by closing the evaporator slit of the helium container and setting the current of the evaporator coil by the toroid transformer. The evaporated gas flows through the holes into the copper tube and gets into the inner tube of the evaporator. The gas heating coil warms the gas to the value set on the electronic temperature regulator. The gas of desired temperature flows through the evaporator and assures good heat exchange with the sample in the quartz heat isolating dewar unit. Finally it is lead from the sample holder unit to the helium collecting network.

The main technical data are the following:

- regulated range of sample temperature:	10...300 K
- temperature stability better than:	$\pm$0.05 K
- maximum liquid helium consumption:	1.5 l/hour
- cubic capacity of helium container:	10 l
- evaporating power:	0...2.5 W
- heating power:	0...5 W

The ESR spectrometer equipped with the low temperature measuring head is used for the investigation of the magnetic properties of highly conducting organic charge trasfer salts. *Figure 2* shows the temperature dependence of the ESR intensity of a $PyTCNQ_2$ single crystal. The ESR intensity is proportional to the paramagnetic susceptibility. In accord with other observations of other physical properties the abrupt jump of the susceptibility around 75 K indicates a phase transition. The phase transition is most likely related to a change of the electronic structure and not much sturctural change is involved. The meas-

urement shows that the magnetic properties are strongly altered around this phase transition. As seen from this example the apparatus, despite its simplicity fulfills the high requirements met in such types of measurements. The apparatus may be used for other applications also /e.g. nuclear magnetic resonance spectroscopy, optical measurements/.

FIGURE CAPTIONS

Fig.1 Scheme of gas-flow measuring head.

Fig.2 Magnetic susceptibility, measured by ESR line intensity, versus temperature of the quasi-one-dimensional charge transfer salt pyridinetetracyanoquinadimethane.

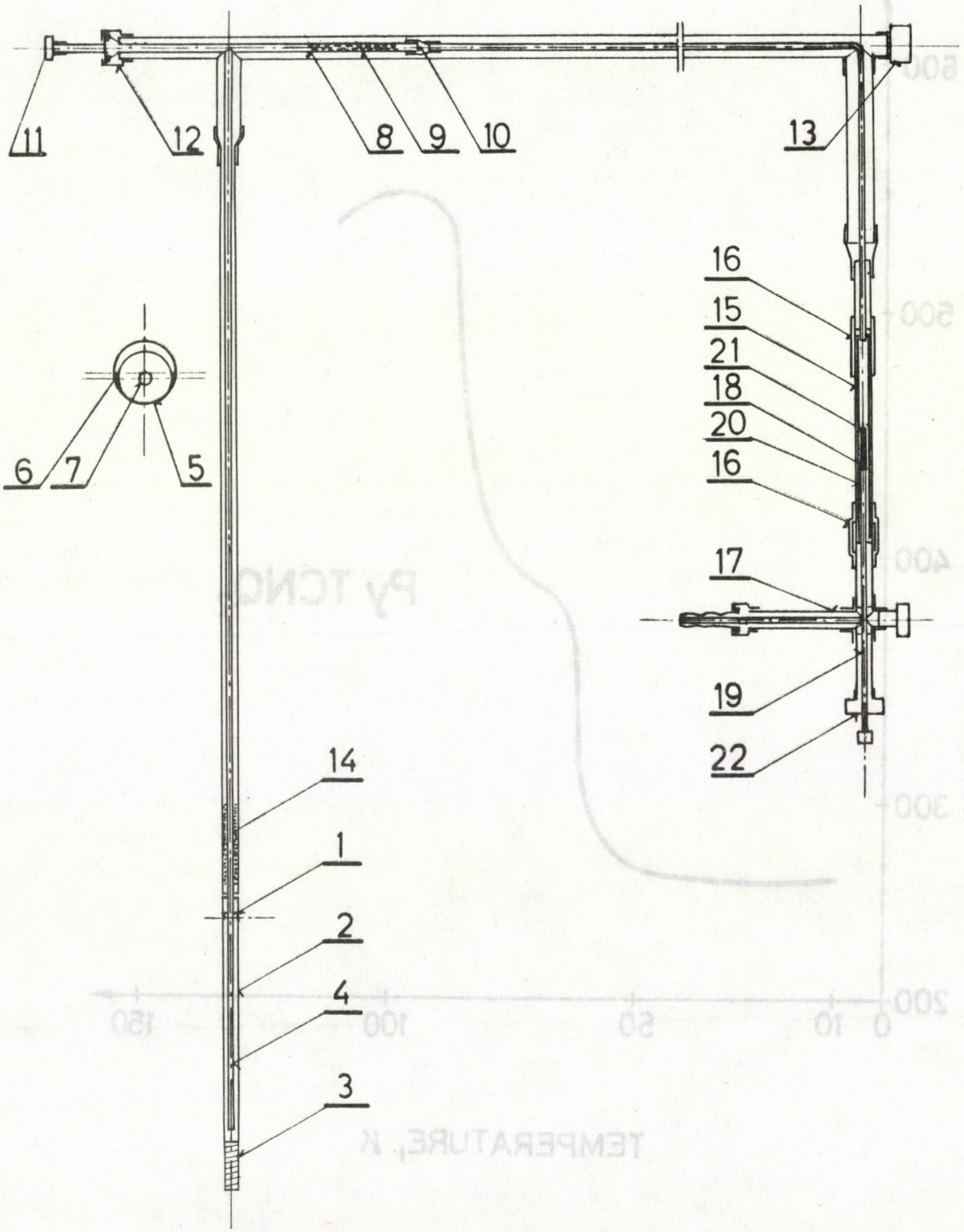


Fig.1

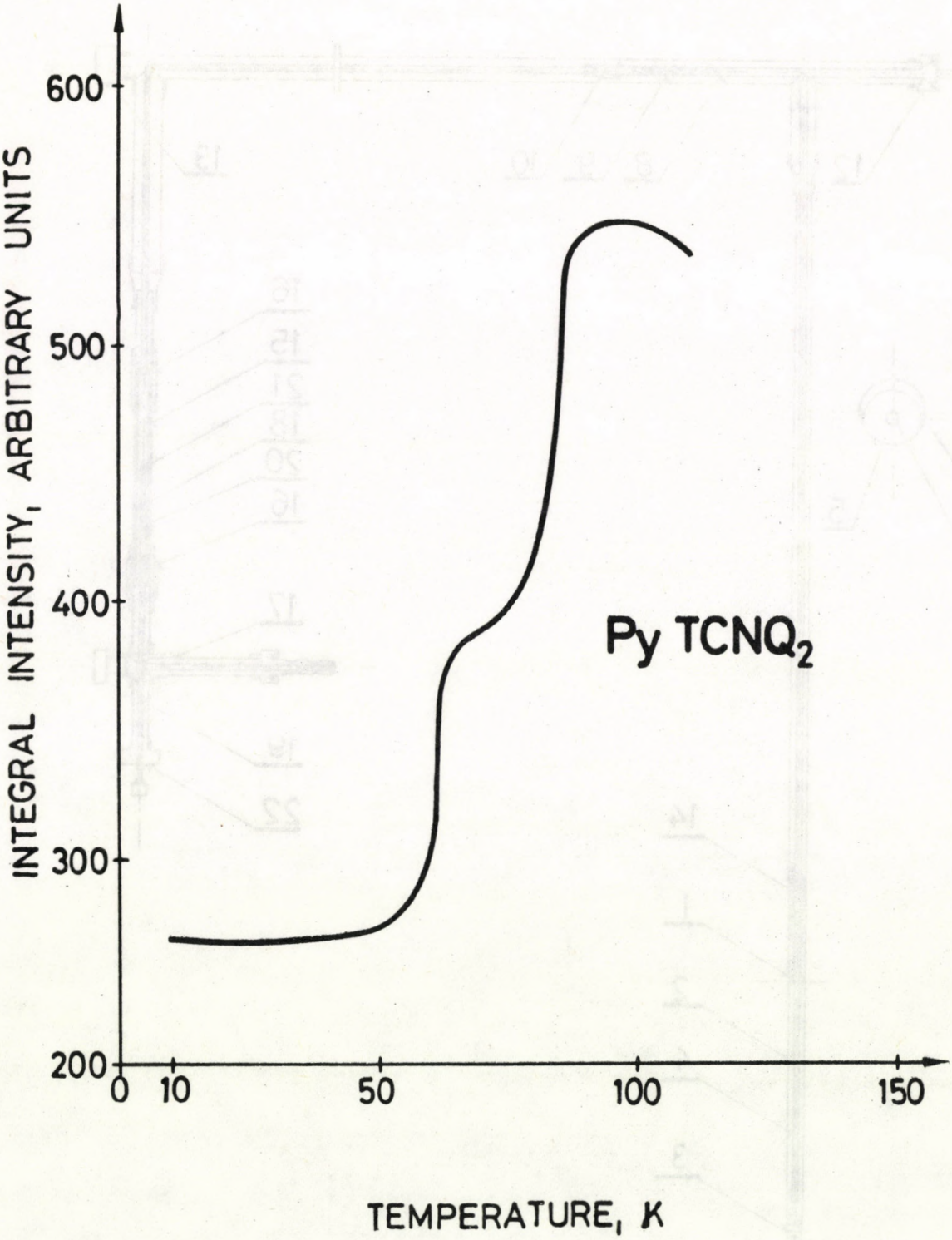
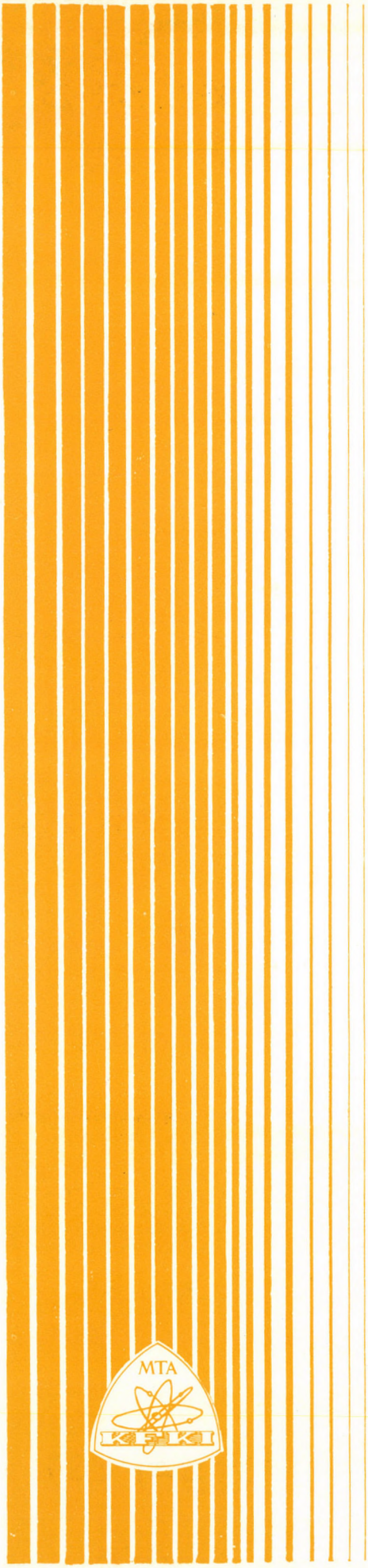

INTEGRAL INTENSITY, ARBITRARY UNITS
600
500
400
300
200
0
10
50
100
150
Py TCNQ2
TEMPERATURE, K

Fig. 2



Kiadja a Központi Fizikai Kutató Intézet
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Szakmai lektor: Grüner György
Nyelvi lektor: Holczer Károly
Példányszám: 55 Törzsszám: 1978-401
Készült a KFKI sokszorosító üzemében
Budapest, 1978. április hó