

# RADIATION AND TEMPERATURE EFFECTS ON THE ELECTRICAL CONDUCTIVITY PROPERTIES OF SOME DISUBSTITUTED ARYLAZO-BARBITURATE AND THIOBARBITURATE COMPOUNDS

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## ABSTRACT

The 5-(disubstituted phenylazo)-barbituric and thiobarbituric acids compounds were prepared. The electrical conductivity properties of these compounds at different temperatures are studied before and after radiation. The  $\sigma_0$ ,  $\Delta E_a$  and  $\phi$  values are determined. The conductivity data are highly affected by the exposure to radiation. Barbituric acid compounds gave some regions to assign changes from semiconducting to insulator behaviour by radiation. 2,5-dimethyl; 2-methyl-4-chloro-and 2, 4-dinitro-thiobarbituric acid compounds, are changed from insulator to semiconducting by radiation. The  $\Delta E_a$  for all barbituric acid compounds except 2-nitro-4-methyl is increased by radiation. At lower temperatures  $\Delta E_a$  values are increased for all thio-compounds except 2, 4-dinitro, while at high temperature the  $\Delta E_a$  values are decreased for 2, 4-dichloro, 2-methyl-4-chloro-and 2-nitro-4-methyl and  $\Delta E_a$  is increased by radiation for 2,5-dichloro;-2, 4-dinitro and 2,5-dimethyl-arylazo thiobarbituric acid compounds. The variation of  $\Delta E_a$  values by radiation is due to creation of new donor levels or of reasonable number of free electrons. The activation energy of charge carrier liberation value,  $\phi$ , depends on the substituent, formation of hydrogen bonding at higher temperature (as in methyl compounds) and quioniond structure at lower temperature (as in nitro-compounds). The mode of conduction is based on a delocclization of  $\pi$ -electrons and the excitation of an electron from highest filled  $\pi$ -molecular orbitals to lowest unfilled one. The relation between  $\log \sigma_0$  and  $\Delta E_a$  is explained.

## INTRODUCTION

Barbiturates and thiobarbiturates are widely used in medicine as hypnotic drugs, especially from their depressive effects on central nervous system [1]. Azo dyes form the largest group of synthetic dyestuffs, due to their widely applications. The azo group involved in a number of important biological reaction such as protein synthesis inhibition, carcinogenesis and azo reduction monoamine oxidase inhibition. Some azo compounds are used for histochemical detection of compounds containing-SH-group [2]. Masoud et al published a series of papers to throw light on the azobarbiturate chemistry [3-21]. In continuation of our

resreach projects, it is interesting to study the effect of radiation on the electrical conductivity of 5-(disubstituted phenylazo)-barbituric and thiobarbituric acids at different temperatures. The organic materials experience show both temporary and permanent changes in properties when subjected to radiation environments. Permanent effects of radiation on organic materials are normally associated with a chemical change in material. Most important among these chemical reactions that occur are molecular associated and cross linkage [22,23].

**Table 1.** Analytical data for 5-(disubstituted phenylazo)-barbituric acid compounds.

Disubstituents	solvent used for crystallization	m.p °C	colour	% Found (% Calculated)		
				C	H	N
2,4-dichloro-	acetic acid	317	dark yellow	39.8 (39.9)	2.2 (2.0)	18.5 (18.6)
2,5-dichloro-	ethanol	300	yellow	39.7 (39.9)	2.5 (2.0)	18.9 (18.6)
2,4-dinitro-	ethanol	314	yellow	37.0 (37.3)	2.0 (1.9)	26.3 (26.1)
2,5-dimethyl-	acetic acid	284	orange-red	48.6 (48.7)	5.3 (5.4)	18.5 (18.9)
2-methyl-4-chloro-	acetic acid	330	yellow	47.6 (47.1)	3.4 (3.2)	19.7 (19.9)
2-nitro-4-methyl-	acetic acid	302	yellow	44.2 (45.4)	3.2 (3.1)	24.3 (24.0)

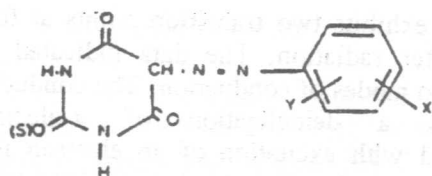
**Table 2.** Analytical data for 5-(disubstituted phenylazo)-thiobarbituric acid compounds.

Disubstituents	solvent used for crystallization	m.p °C	colour	% Found (% Calculated)		
				C	H	N
2,4-dichloro-	dioxane	328	yellow	38.0 (37.9)	2.2 (1.9)	17.4 (17.7)
2,5-dichloro-	ethanol	260	yellow	39.0 (37.9)	2.0 (1.9)	17.8 (17.7)
2,4-dinitro-	ethanol	250	orange	36.0 (35.5)	2.0 (1.8)	24.1 (24.8)
2,5-dimethyl-	acetic acid	283	red	52.2 (52.5)	4.4 (4.7)	20.3 (20.4)
2-methyl-4-chloro-	ethanol	291	yellow	44.9 (44.5)	3.4 (3.1)	18.5 (18.9)
2-nitro-4-methyl-	acetic acid	above 292	yellow	44.6 (43.0)	3.2 (3.0)	23.2 (22.8)

## EXPERIMENTAL

The compounds (I) were prepared by a diazotization process [24] of barbituric or thiobarbituric acid. The amines used were 2,4-dichloro-; 2,5-dichloro-; 2,4-dinitro-; 2,5-dimethyl-; 2-nitro-4-methyl- and 2-methyl-4-chloro-anilines. The CHN analytical data of the compounds carried out at the central

microanalytical laboratory of Alexandria University are collected in Tables (1) and (2). The solvents used for crystallization of compounds were collected in Tables (1) and (2). These samples were washed with deionized water to remove traces of ions.



X	Y
2-Cl	4-Cl
2-Cl	5-Cl
2-NO <sub>2</sub>	4-NO <sub>2</sub>
2-CH <sub>3</sub>	5-CH <sub>3</sub>
2-NO <sub>2</sub>	4-CH <sub>3</sub>
2-CH <sub>3</sub>	4-Cl

The d.c electrical conductivities were conducted using a circuit which schematically represented in Figure (1), as previously reported by Owen [25]. The electrical conductivity of the compounds was measured in the temperature range 303-434 °K. The sample was prepared in the form of a tablet at a pressure of 4 ton cm<sup>2</sup>. The tablet was 12mm in diameter at 0.1-0.3mm in thickness. The sample was held between two copper electrodes with silver paste inbetween, and then inserted with the holder vertical into a cylindrical electric furnace. Both ends of furnace were closed off to reduce draughts. The potential drop across the heater was varied gradually through a variac transformer to produce a slow rate of the increase of temperature. This leads to accurate temperature measurements. The circuit used to measure the electrical conductivity consisted of d.c regulated power supply (Heathkit, 0 to 400 v) and a Keithley multimeter for measuring current with a sensitivity of 10<sup>-9</sup>. The temperature of the sample was measured within ±0.1 °K by means of copper-constantan thermocouple. The conductivity,  $\sigma$ , was recorded through raising the temperature using the general equation:-

$$\sigma = \frac{I d}{V_e a}$$

where I is the current in amps and V<sub>c</sub> is the potential drop across the sample of cross-section area, a, and thickness, d. A neutron beam of an energy of  $\cong 14.5$  MeV and flux  $5.8 \times 10^5$  cm<sup>-2</sup> Sec<sup>-1</sup> was used with exposure for 12h irradiation. The electrical conductivity measurements were done for a sufficiently long time to allow the decay of polarization effects.

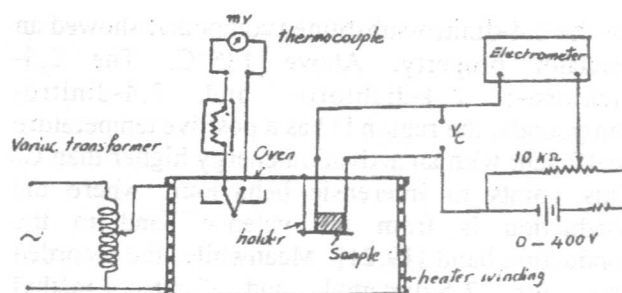


Figure 1.

## RESULTS AND DISCUSSION

The dependence of the electrical conductivity of semiconducting materials on temperature is expressed by the following equation:  $\sigma = \sigma^* e^{-\Delta E/2Kt}$  where  $\Delta E$  is the activation energy for conduction,  $\sigma^*$  is a constant for the conductivity independent of temperature and k is the Boltzmann constant. The electrical conductivity data are represented in Figures (2) & (3) and collected in Tables (3) and (4) before and after radiation. The followings are given:

- A) The temperature dependence of the conductivity curves of all the barbituric acid series except 2, 5-dimethyl- and 2-nitro-4-methyl-phenylazobarbituric acids showed four regions (A,B,C and D) before radiation. On exposure to radiation, the compounds gave variable regions. Both the 2,5-dimethyl- and 2-nitro-4-methyl compounds exhibit two regions before and after radiation.
- B) The regions (A&B) are with -ve temperature coefficient of electrical conductivity while the regions (C&D) are with +ve values. In the temperature range 30-44°C and 44-48°C, the regions A and B appeared for 2,4-dichloro and 2,4-dinitro-compounds before radiation. However, the 2,5-dichloro compound gave both regions independent of radiation. These exhibit a decrease of conductivity as the temperature is increased due to scattering of carriers by photons due to lattice vibrations within the temperature range studied. In general  $\Delta E_B > \Delta E_A$  above 84°C, the conductivity of these compounds revealed positive values in regions C and D. The region C for the 2,5-dichloro and 2,4-dichloro substituted compounds showed a low activation energy accompanied with an extrinsic behaviour [19]. However, the region C

for the 2,4-dinitro-substituted compound showed an insulator property. Above 135°C, The 2,5-dichloro-; 2,4-dichloro- and 2,4-dinitro-compounds, the region D has a positive temperature coefficient with an activation energy higher than C. This points to interinsic behaviour, where the conduction is from the valence band to the conduction band [19,21]. Meanwhile, the recorded data for 2,5-dimethyl and 2-nitro-4-methyl-derivatives, Figure 2, showed only two regions (A&D). The absence of B&C regions are more likely due to the width of the forbidden gap [19]. The shallow behaviour within the temperature range (44-84°C) and (84-127°C) including regions B and C, respectively, for 2,5-dichloro before and after radiation and (2,4-dichloro and 2,4-dinitro) before radiation can be attributed to the thermal keto  $\rightleftharpoons$  enol tautomerism [26]. Therefore, it would be reasonable to assume that conduction process is mainly initiated through delocalization of  $\pi$ -electrons, followed by excitation of an electron from the highest field  $\pi$ -molecular orbitals to the lowest unfilled ones [27]. The absence of the shallow behaviour for the 2,5-dimethyl and 2-nitro-4-methyl-substituted compounds indicated their existence in only one geometrical form (keto or enol). The, IR spectra of the barbituric acid compounds under investigation are convenient with the conductivity behaviour. The temperature-conductivity curves for thiobarbituric acid compounds containing, 2,4-dichloro-; 2,5-dichloro- and 2,4-dinitro-groups reveal three regions A,B, and C. How ever, the 2-methyl-4-chloro-; 2,5-dimethyl- and 2-nitro-4-methyl- compounds exhibit two regions (A&C), Fig.3. Certainly, this is due to the electronic character of the substituent. The region A is characterised by the decrease of conductivity as the temperature is increased. The region C has a positive temperature coefficient with activation energy higher than that of region A, to assign the semiconducting properties. Two modes of conduction with two activation energy values,  $\Delta E_A$  and  $\Delta E_C$  are given. Also, the conductivity-temperature curves, Figure (3)., reveal two transition points for 2,4-dichloro-; 2,5-dichloro- and 2,4-dinitro- before radiation at 79.1, 101.5; 74.2, 94.6 and 60.3, 75.4°C. These become at 54.8, 101.5; 84.1; 100,13 and 74.2, 104.3°C after radiation, respectively. Also the 2,5-dimethyl compound showed two transition points at 65.9; 68.7°C before radiation. The 2-methyl- 4- chloro

-compound exhibits two transition points at 63.7; 104.3°C after radiation. The data indicated that there are two modes of conduction. The conduction process is a delocalization of  $\pi$ -electrons accompanied with excitation of an electron from highest filled  $\pi$ -molecular orbitals to lowest unfilled ones [27].

The region B has insulator character before and after radiation for 2,5-dichloro-; 2,4-dinitro- before radiation and 2-methyl-4-chloro-after radiation. The region B is valued, Table (4), for 2,4-dichloro, 2,5-dimethyl- and 2,4-dinitro compounds; before and after radiation respectively. The region B is due to the transition between A and B regions.

In general, the phenomenon of discontinuation observed in the electrical conductivity-temperature relationship- for 5-(disubstituted phenylazo)-barbituric and thiobarbituric acid compounds-showed more than segment, Tables (3&4), with variable activation energies, probably due to the presence of different crystallographic or phases transitions [21].

It seems that the electrical conductivity properties of the compounds is highly affected by the exposure to radiation.

- i) For the barbituric acid series, Figure (2).: 2, 5-dimethyl-barbituric acid ligand, the conductivity in the temperature range (30-84°C) changed from semiconductor before radiation to insulator after radiation. Below 43°C, region A disappeared by radiation for 2,4-dinitro-barbituric acid compound and insulator behaviour (region C) appeared including large area by radiation. However, the semiconducting behaviour in regions (C&D) before radiation becomes insulator character after radiation for 2,4-dichloro-derivative. On the other hand, no change in the conductivity temperature diagram for 2,5-dichloro-barbituric acid except shift in temperature range for different regions i.e the shallow behaviour exists after and before radiation due to the presence of thermal keto-enol tautomerism [26] before and after radiation. However, the thermal-keto-enol tautomerism disappeared by radiation for 2,4-dichloro-and 2,4-dinitro-barbituric acid compounds.
- ii) The radiation exposure affects the electrical conductivity properties of thiobarbituric acid compounds. The region B is absent in the substituted 2,5-dimethyl compound. The region B appeared with insulator property and changed from insulator to semiconducting property with

respect to 2-methyl-4-chloro and 2,4- dinitro respectively.

- iii) In general, the decrease in electrical conductivity by radiation may be attributed to the creation interstitial anions and cations [28]. These defects may be result in dissociation and/or recombination of different anions and cations beside the breakdown of chemical bonds.
- iv) In all barbituric acid series except 2-nitro-4-methyl compound, the activation energies increased by radiation. However, for the thio-compounds, the activation energy for region A is increased by radiation for all compounds except 2,4-dinitro compound. While in region C (at higher temperature) the activation energy is increased by radiation for 2,5-dichloro; 2,4 dinitro-; 2,5-dimethyl and decreased for 2,4-dichloro; 2-methyl-4-chloro-and 2-nitro-4-methyl-phenylazo thiobarbituric acid compounds. The decreasing in  $\Delta E_a$  may be attributed to that the radiation introduces doner centers [29] and/or enhances ionic association in solids. Thus, it can be stated [30] that the exposure to radiation leads to an increase in the concentration of existing levels or creating new doner levels. However, the increasing of activation energies is probably due to action of ionizing absorbed gamma dose on the creation of reasonable number of free electrons even at room temperature, simulating the free electrons.

The activation energy for the process of carrier liberation [31] could be calculated as follow:

$$\phi = \phi_2 - \phi_1$$

where  $\phi_2$  and  $\phi_1$  are the activation energy for conduction after and before radiation, respectively. The activation energies of carrier liberation ( $\phi$ ) for compounds are collected in Tables (3 & 4). The data give the following observations:

- i) The  $\phi$  values for 2,4-dichloro-phenylazo-barbituric and thiobarbituric acids compounds in region A are greater than the corresponding 2,5 -dichloro-derivatives. The investigation of region D for the dichloro-barbituric acid and region C for the dichloro-thiobarbituric acid compounds reserved trend. This behaviour may be attributed to the mesomeric effect of -Cl- group in-p- and -m-positions.

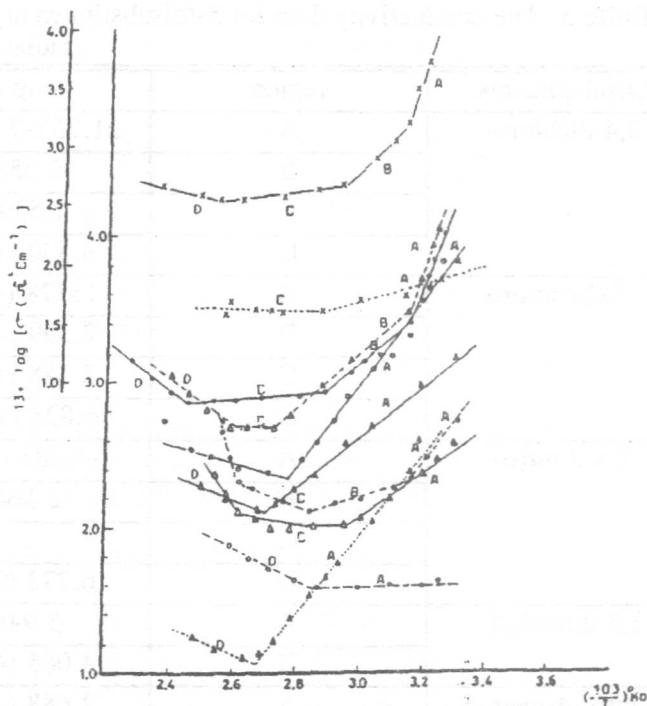


Figure 2. Electrical conductivity of 5-(disubstituted arylazo)-barbituric acid compounds as a function of temperature before (—) and after (---) radiation  
 (xxxx) 2,4-dichloro- (o o o o) 2,5-dimethyl-  
 (.....) 2,5-dichloro- (.....) 2-nitro-4-methyl-  
 (.....) 2,4-dinitro-

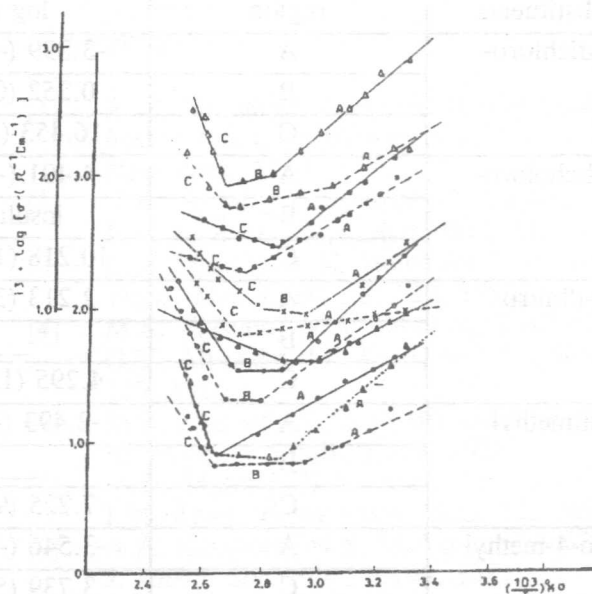


Figure 3. Electrical conductivity of 5-(disubstituted arylazo)-thiobarbituric acid compounds as a function of temperature before (—) and after (---) radiation.  
 (.....) 2,4-dichloro- (xxxx) 2,5-dimethyl-  
 (o o o o) 2,5-dichloro- (.....) 2-methyl-4-chloro-  
 (.....) 2,4-dinitro- (.....) 2-nitro-4-methyl-

**Table 3.** The conductivity data for 5-(disubstituted arylazo)-barbituric acid compounds before and (after) radiation.

Disubstituents	region	$\log \sigma_0$	$\Delta E_a$	$\phi$
2,4-dichloro-	A	-21.76 (-7.66x10 <sup>-1</sup> )	-1.578 (-0.163)	1.415
	B	-5.658 (-)	-0.114 (-)	
	C	1.539 (-)	0.0861 (-)	
	D	3.830 (1.854)	0.099 (0.0151)	-0.0839
2,5-dichloro-	A	-15178 (-7.186)	-1.171 (-0.595)	0.576
	B	-2.399 (-7.155)	-0.361 (-0.132)	0.229
	C	2.388 (1.856)	0.036 (0.163)	0.127
	D	6.828 (18.835)	0.322 (1.251)	0.929
2,4-dinitro-	A	-14.383 (-1.614)	-1.089 (-0.243)	0.846
	B	-2.566 (-)	-0.380 (-)	-
	C	—	insulator	-
	D	6.772 (9.145)	0.311 (0.561)	0.250
2,5 dimethyl-	A	-5.948 (-)	-0.594 (insulator)	-
	D	4.065 (4.728)	0.124 (0.224)	0.100
2-nitro-4-methyl	A	-2.688 (-6.062)	-0.351 (-0.533)	-0.182
	D	4.384 (3.114)	0.1697 (0.151)	-0.0187

**Table 4.** The conductivity data for 5-(disubstituted arylazo)-thiobarbituric acid compounds before and (after) radiation.

Disubstituents	region	$\log \sigma_0$	$\Delta E_a$	$\phi$
2,4-dichloro-	A	-3.399 (-2.218)	-0.382 (-0.264)	0.118
	B	0.252 (0.414)	-0.244 (-0.103)	0.141
	C	16.453 (8.545)	1.078 (0.504)	-0.574
2,5-dichloro-	A	-4.401 (-3.049)	-0.410 (-0.307)	0.103
	B	insulator	insulator (insulator)	
	C	10.218 (11.394)	0.594 (0.767)	0.173
2,4-dinitro-	A	2.213 (3.477)	-0.254 (-0.356)	0.106
	B	—	insulator (0.0495)	
	C	4.295 (15.740)	0.184 (1.109)	0.925
2,5 dimethyl-	A	-2.493 (1.639)	-0.297 (-0.059)	0.239
	B	—	0.0975 (-)	
	C	7.225 (9.369)	0.374 (0.546)	0.172
2-nitro-4-methyl	A	-3.546 (-2.116)	-0.345 (-0.248)	0.101
	C	3.739 (5.177)	0.148 (0.266)	0.126
2-methyl-4-chloro-	A	-1.083 (-0.955)	-0.222 (0.198)	0.024
	B	(-)	(insulator)	
	C	16.76 (9.413)	1.116 (0.554)	-0.559

- ii) The  $\phi$  2,4-dinitro >  $\phi$  2,5-dimethyl >  $\phi$  2-nitro-4-methyl compound in region D for barbituric acid and in region C for thiobarbituric acid compounds. Based on region A for thiobarbituric acid compounds the following order is given:  $\phi$  2,5-dimethyl >  $\phi$  2-nitro-4-methyl >  $\phi$  2,4-dinitro. Two main factors are responsible for such behaviour: i) the existence of an intramolecular hydrogen bond between O-CH<sub>3</sub> and N=N groups at higher temperature (see regions C&D) and the formation of quioniond structure for the nitro-compounds at lower temperature (see region A).
- iii) The  $\phi$  values have the following sequence for the thio-series: 2,5-dimethyl > 2,4-dichloro > 2,5-dichloro > 2-methyl-2-chloro-. Generally, both the substituents and the doses of radiation are strongly responsible for the creation of free charge carriers. The low values of  $\phi$  of the thio-compounds compared to the oxygen homologous could be attributed to the electronegativity difference between oxygen and sulphur atoms. So the number of creation sites of the thio-compounds is smaller than of oxygen homologous

Figure 4 represents the relation between  $\Delta E_a$  and  $\log \sigma_o$ . Best fit straight lines are obtained. The following empirical equations are deduced

**For the barbituric acid series:**

- region A:  $\Delta E_a = 0.064 \log \sigma_o = -0.17$  (before radiation)  
 $\Delta E_a = 0.071 \log \sigma_o = -0.11$  (after radiation)
- region D:  $\Delta E_a = 0.084 \log \sigma_o = -0.225$  (before radiation)  
 $\Delta E_a = 0.088 \log \sigma_o = -0.11$  (after radiation)

**For the thiobarbituric acid series:**

- region A:  $\Delta E_a = 0.057 \log \sigma_o = -0.157$  (before radiation)  
 $\Delta E_a = 0.053 \log \sigma_o = -0.150$  (after radiation)
- region C:  $\Delta E_a = 0.088 \log \sigma_o = -0.150$  (before radiation)  
 $\Delta E_a = 0.083 \log \sigma_o = -0.160$  (after radiation)

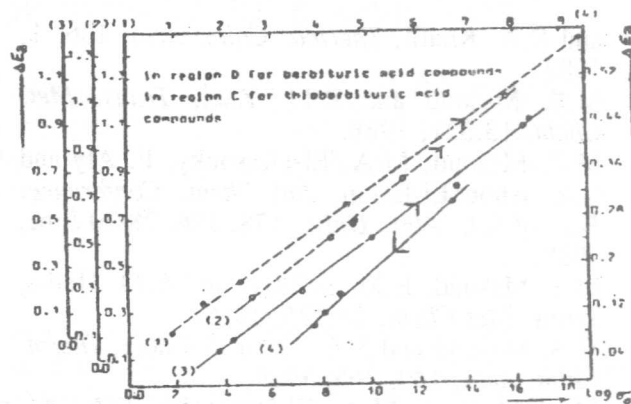
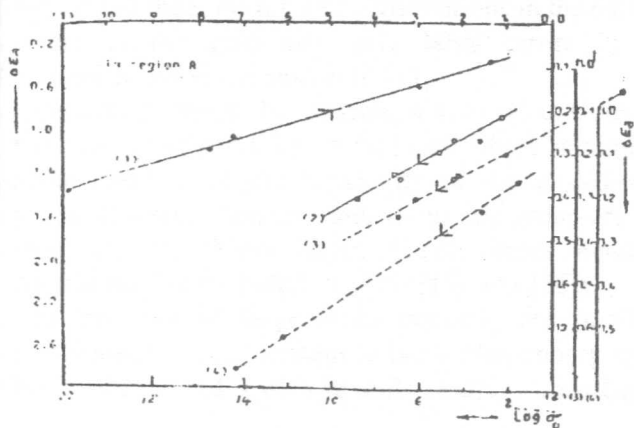


Figure 4. The relation between  $\Delta E_a$  and  $\log \sigma_o$  before (---) and after (- - -) radiation for barbituric (...) and thiobarbituric (ooo) acid compounds.

**LIST OF SYMBOLS**

- I is the current in ampers
- k is the Boltzman constant
- T is the absolute temperature °K
- V<sub>c</sub> is the potential drop across the sample
- $\Delta E_a$  is the activation energy for conduction
- $\sigma$  is the electrical conductivity
- $\sigma^*$  is a constant for the conductivity independent of temperature
- $\phi$  is the activation energy of carrier liberation

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