A *trans*-Coplanar Elimination of Sulfur Dioxide and Chloride Ion in 2-Chloroalkanesulfinates*

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2-Chloroalkanesulfinates are shown to decompose into alkenes, sulfur dioxide, and chloride ions. The mechanism of the reaction is discussed. The reaction is shown to proceed *via* a *trans*-coplanar elimination. The possible implication of a 2-substituted alkanesulfinate in the base-promoted rearrangement of α-halosulfones (Ramberg-Bäcklund rearrangement) is discussed.

The decomposition of a 2-haloalkanesulfinate has not previously been studied. However, it is known [1] that the barium salt of 2-hydroxyethanesulfinic acid decomposes to yield ethylene and barium sulfite. During the course of this work the decomposition of perfluoroalkanesulfinates to yield perfluoroalkenes has been reported.[2] It is also of interest to note that a 2-halosulfinate has been discussed [3] as an intermediate in the base-induced rearrangement of α-halosulfones to alkenes (Ramberg-Bäcklund rearrangement).

In connection with a study of sulfur dioxide elimination reactions, 2-chloroethanesulfinic acid was treated with base. The sulfinate was found to decompose spontaneously to ethylene, chloride ion, and sulfur dioxide in good yield (Scheme I). In this paper the decomposition of 2-chloroalkanesulfinates will be discussed and evidence for a *trans*-coplanar elimination will be presented.

2-Chloroethanesulfinate (*1*) is readily available and has been used as a model compound for kinetic and mechanistic studies. This compound was prepared from the corresponding sulfinyl chloride which may be prepared by addition of thionyl chloride to ethylene according to a previous method.[4] A more convenient method for the preparation of 2-chloroethanesulfinyl chloride has now been developed. Treatment of ethylene sulfide and acetic anhydride in equivalent amounts with chlorine afforded 2-chloroethanesulfinyl chloride in good yield. The corresponding sulfinic acid was prepared by a careful hydrolysis with aqueous sodium hydrogen carbonate followed by acidification with sulfuric acid. The 2-chloroethanesulfinic acid was extracted with ethyl ether. The crude sulfinic acid, obtained after evaporation of the solvent, was found to be pure and was characterized by IR and NMR.

The decomposition of 2-chloroethanesulfinate (*1*) can be performed in water or in organic solvents. The rate of the decomposition in water with excess sodium hydroxide has been determined. The formation of ethylene was found to follow first order kinetics and the activation energy of the reaction was 127.8 ± 8.4 kJ mol^{-1}.
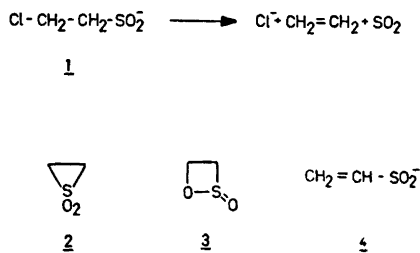
Scheme 1.

Different reaction mechanisms for the decomposition reaction can be formulated. Ethylene sulfone (2) can be ruled out as an intermediate since this compound was found to be stable under the reaction conditions used. A less probable intermediate is 1,2-oxathietane 2-oxide (3, β-sultine). β-Sultines are known [5] to decompose to alkanes and sulfur dioxide. However, they also react with alcohols to give 2-hydroxyalkanesulfinic acid esters.[5]

When the decomposition of 2-chloroethanesulfinate was carried out in methanol in the presence of triethylamine, no methyl 2-hydroxyethanesulfinate could be detected in the reaction product.

When the decomposition of 2-chloroethanesulfinate was performed in a deuterium oxide solution, no deuterium incorporation could be detected in the ethylene as shown by the mass spectrum of the 1,2-dibromoethane formed by bromination of the ethylene. Thus ethylenesulfinate (4) derived from the 2-chloroethanesulfinate by hydrogen chloride elimination, cannot be an intermediate in the reaction.

The most likely mechanism for the 2-halosulfinate decomposition is a trans-coplanar elimination of sulfur dioxide and chloride ion. In order to obtain additional information regarding this reaction its stereochemistry was investigated as follows.

The addition of sulfur monochloride to alkenes is known to proceed via a trans-addition yielding the corresponding 2-haloalkyl disulfides [6] (Scheme 2). Lautenschlaeger and Schwartz have recently shown [6] that the addition to cis-2-butene yields bis(threo-2-chloro-1-methylpropyl)disulfide (threo-5) which upon treatment with sodium sulfide gives the cis-2,3-dimethylthiirane (6). The corresponding trans-2-butene yields the trans-2,3-dimethylthiirane under similar conditions.

We have reacted cis-2-butene with sulfur monochloride. The disulfide (threo-5) thus obtained was oxidized with chlorine in acetic anhydride to yield the corresponding threo-2-chloro-1-methylpropanesulfinyl chloride (threo-7) by a known reaction.[7] This compound was hydrolysed with aqueous sodium hydroxide. The intermediate threo-2-chloro-1-methylpropanesulfinate (threo-8) rapidly decomposed to yield cis-2-butene. Less than 1 % trans-2-butene was present in the product as shown by gas chromatography.

Similarly trans-2-butene yielded erythro-2-chloro-1-methylpropanesulfinyl chloride (erythro-7) which, without isolation, was treated with aqueous sodium hydroxide to yield trans-2-butene via the intermediate erythro-2-chloro-1-methylpropanesulfinate (erythro-8). Less than 1 % cis-2-butene was present in the product.
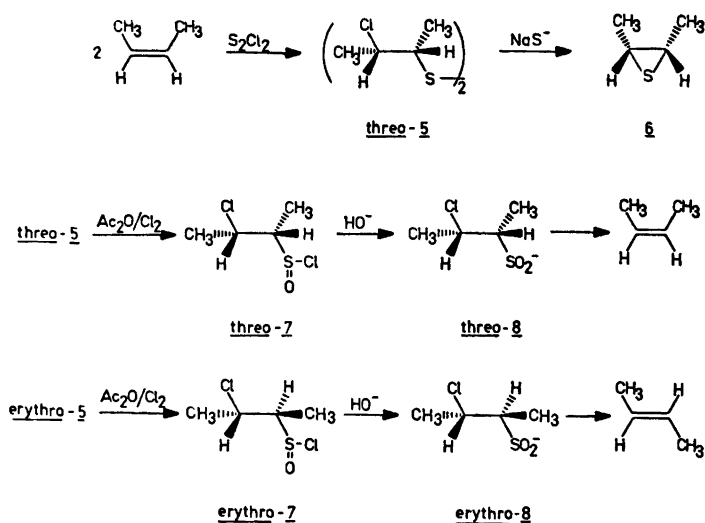
Scheme 2.

The 2-halosulfinate decomposition is thus a stereospecific reaction and the results presented above support a mechanism involving a *trans*-coplanar elimination.

A 2-chloroalkanesulfinate decomposition also occurs with 2,2,2-trichloroethanesulfinate (*9*, Scheme 3). When the sulfinyl chloride of *9* was hydrolysed with an equivalent amount of aqueous sodium hydroxide, 1,1-dichloroethylene (*12*) was formed. With an excess of sodium hydroxide, chloroacetylene (*13*) was isolated in addition to dichloroethylene (*12*). Two possible routes for the formation of chloroacetylene may be considered. In one the 2,2-dichloroethylene-sulfinate intermediate (*10*) decomposes *via* a reaction mechanism similar to that for the 2-halosulfinate and in the other, further de-hydrohalogenation of the 2,2-dichloroethylene-sulfinate (*10*) gives 2-chloroacetylenesulfinate (*11*) which decomposes into chloroacetylene (*13*) and sulfur dioxide.[8]
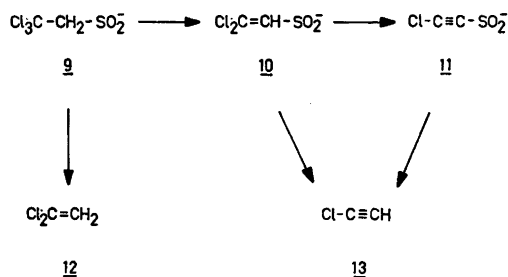
Scheme 3.

There is strong evidence for the presence of intermediate thiirane 1,1-dioxides in the base-induced rearrangement of α-halosulfones (Ramberg-Bäcklund rearrangement).[9] The decomposition of this intermediate thiirane 1,1-dioxide to alkene and sulphur dioxide is a stereospecific reaction [9] (Scheme 4). Although this decomposition is known to be a thermal reaction it is not clear whether the base, in some cases, may take part in the decomposition. It has been established that ethylene sulfone upon treatment with alkali yields 2-hydroxyethanesulfinate.[1] Hence nucleophilic attack of the base on the intermediate thiirane 1,1-dioxide in the Ramberg-Bäcklund rearrangement should yield 2-substituted alkane-sulfinates. This reaction has been discussed by

Bordwell *et al.*[10] They propose that an inter-mediate 2-methoxyalkanesulfinate would appear to be stable under conditions whereby thiirane 1,1-dioxides are converted to alkenes. Therefore, they conclude that this mechanism does not operate in the decomposition of thiirane 1,1-dioxides in the presence of methoxide ions. However, there are no indications that alkane-sulfinates with a leaving group in 2-position are stable under the conditions for a Ramberg-Bäcklund rearrangement and the formation of an intermediate 2-substituted alkanesulfinate cannot be excluded. This intermediate would then decompose by a reaction analogous to the stereospecific decomposition of 2-chloroalkane-sulfinates (Scheme 4).
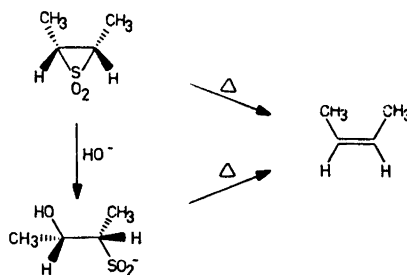
Scheme 4.

EXPERIMENTAL

All boiling points are uncorrected. IR spectra were measured on a Perkin Elmer Model 421 infrared spectrophotometer. NMR spectra (TMS internal standard) were recorded on a Varian Model A-60A instrument. Mass spectra were obtained using an LKB Model 9000 mass spectrometer.

2-Chloroethanesulfinyl chloride. A solution of ethylene sulfide (23.5 g, 0.39 mol) and acetic anhydride (40 g, 0.39 mol) was cooled to 5 °C. Chlorine was then passed into the solution at such a rate that the temperature remained between 10 and 15 °C. When no more chlorine was consumed excess chlorine and acetyl chloride was removed by evaporation *in vacuo* at room temperature. The residue was distilled at reduced pressure to give 2-chloroethane-sulfinyl chloride; b.p. 42 °C/0.7 mmHg (lit.[4] b.p. 81 °C/9 mmHg); yield 46 g (80 %); IR (film) 1150 cm^{-1} (S=O); NMR (CDCl$_3$) δ 3.95 ppm (m, CH$_2$).

2-Chloroethanesulfinic acid. 2-Chloroethane-sulfinyl chloride (14.7 g, 0.1 mol) was slowly added to an aqueous solution (100 ml) of sodium hydrogen carbonate (16.8 g, 0.2 mol) cooled to 5 °C. The mixture was stirred for 10 min and

then acidified with conc. sulfuric acid (10 g). The 2-chloroethanesulfinic acid thus formed was extracted with ethyl ether (200 ml). The solution was dried and the solvent evaporated *in vacuo* to give the oily 2-chloroethanesulfinic acid; yield 9 g (70 %); IR (film) 1080 (broad) cm^{-1} (SO$_2$H); NMR (CDCl$_3$) δ 3.2 (t, 2, CH$_2$) and 3.9 ppm (t, 2, CH$_2$).

Decomposition of 2-chloroethanesulfinic acid in the presence of sodium hydroxide. 2-Chloroethanesulfinic acid (8 g, 0.062 mol) was added to an aqueous sodium hydroxide solution (2 M, 100 ml). The reaction mixture was slowly heated to 60 °C and was then kept at that temperature for 10 min. The ethylene thus formed was passed through a chloroform solution (100 ml) containing bromine (16 g, 0.1 mol). The chloroform solution was treated with an aqueous sodium bisulfite solution (10 %) in order to remove unreacted bromine, washed with water and dried. Evaporation of the solvent afforded crude 1,2-dibromoethane; b.p. 131 − 132 °C/760 mmHg; yield 4 g (34 %); n_D^{22} 1.5360. The 1,2-dibromoethane was analysed by GLC-MS (column, 150 cm × 3 mm, packed with 5 % SE 30 on Chromosorb W 60/80 mesh, column temperature 70 °C), *m/e* (rel. intensity); C$_2$H$_4$Br$^+$ 107(100), 109(100); C$_2$H$_4$Br$_2$$^+$· 186(1.3), 188(2.6), 190(1.3).

Decomposition of 2-chloroethanesulfinyl chloride in the presence of D$_2$O/NaOD. 2-Chloroethanesulfinyl chloride (1 g, 6.8 mmol) was added to a sodium deuterium oxide solution (1 M, 15 ml) in deuterium oxide. The mixture was heated to ensure decomposition of 2-chloroethanesulfinate. The ethylene gas was passed through a chloroform solution containing bromine to give 1,2-dibromoethane. The excess of bromine was removed in the usual way. The 1,2-dibromoethane was analysed by GLC-MS as has been described above. No deuterated 1,2-dibromoethane could be detected in the reaction mixture. The mass spectrum of the product was identical with that of an authentic sample of 1,2-dibromoethane.

Decomposition of 2-chloroethanesulfinic acid in the presence of ethylene sulfone. The NMR spectrum of 2-chloroethanesulfinic acid in chloroform solution exhibits peaks at δ 3.2 (t, 2, CH$_2$), 3.9 (t, 2, CH$_2$) and 7.4 ppm (s, SO$_2$H). When excess pyridine is added to the sulfinic acid, gas is evolved. The peaks at δ 2.93 and 3.85 ppm of 2-chloroethanesulfinate decrease. 2-Chloroethanesulfinate disappeared after 18 min and no other peaks except that of ethylene (δ 5.22 ppm) could be detected in this region. Ethylene sulfone[11] (δ 3.12 ppm) was found to be stable in pyridine/chloroform solution under the condition used. No appreciable decrease of the peak at δ 3.12 ppm was observed after 1 h. 2-Chloroethanesulfinic acid and ethylene sulfone were mixed in a chloroform solution. Excess pyridine is then added and the decomposition of 2-chloroethanesulfinate is followed by NMR. When the 2-chloroethane-

sulfinate is completely consumed (*ca.* 20 min) the intensity of the peak from the ethylene sulfone (δ 3.12 ppm) is still undiminished.

Decomposition of 2-chloroethanesulfinate in the presence of methanol. Triethylamine (2 g, 0.02 mol) was added to a solution of 2-chloroethanesulfinic acid (2.5 g, 0.02 mol) in methanol (40 ml) at room temperature. The reaction mixture was then boiled for 30 min. The presence of sulfur dioxide could be detected by the characteristic smell of the gas. The volatile constituents in the solution were then evaporated *in vacuo* to give a crystalline residue which was washed several times with ethyl ether. The combined ether solutions were then washed with water and dried. Evaporation of the solvent under reduced pressure did not afford any residue. The insoluble crystals which did not dissolve in ethyl ether was characterized as triethylammonium chloride which upon treatment with alkali gave triethylamine. This experiment shows that methanol does not react with any intermediate (*e.g.* β-sultine) during the decomposition of 2-chloroethanesulfinate and it is thus unlikely that any such intermediate is involved in the decomposition reaction.

Bis(2,2,2-trichloroethyl)disulfide was prepared essentially according to the method of Aichenegg.[13] Sulfur monochloride (81 g, 0.6 mol) was added dropwise over 2 h to a mixture of 1,1-dichloroethylene (250 g, 2.58 mol) and FeCl$_3$ (8 g). The reaction mixture was kept at room temperature overnight and was then diluted with 1 vol of acetone, followed by addition of sufficient water to give 2 layers. The organic phase was recovered and washed with water. The procedure was repeated until no more acetone was present in the organic phase, which was then dried and evaporated *in vacuo* to give a residue. Distillation at reduced pressure afforded bis(2,2,2-trichloroethyl)disulfide; b.p. 122 °C/0.5 mmHg (lit.[12] b.p. 145 − 150 °C/0.36 − 0.46 mmHg); yield 90 g (46 %); n_D^{25} 1.5750 (lit.[12] n_D^{20} 1.5780).

2,2,2-Trichloroethanesulfinyl chloride. A solution of bis(2,2,2-trichloroethyl)disulfide (32.8 g, 0.1 mol) and acetic anhydride (20.4 g, 0. 2 mol) was cooled to 5 °C. Chlorine was then passed into the solution at such a rate that the temperature remained between 10 and 15 °C. When no more chlorine was consumed, chlorine and acetyl chloride were removed by evaporation *in vacuo* at room temperature. The residue was distilled at reduced pressure to give 2,2,2-trichloroethanesulfinyl chloride; b.p. 56 °C/1.5 mmHg; yield 32 g (74 %); n_D^{25} 1.5446; IR (film) 1160 cm^{-1} (S=O); NMR (neat) δ_A 4.68 and δ_B 4.85 ppm (J_{AB} 14 Hz).

Decomposition of 2,2,2-trichloroethanesulfinyl chloride with an equivalent amount of base. An aqueous sodium hydroxide solution (1.6 M, 50 ml) was slowly added to a cooled mixture (5 °C) of 2,2,2-trichloroethanesulfinyl chloride (8.6 g, 0.04 mol) and water (150 ml). Carbon tetrachloride (50 ml) was then added to dissolve

the 1,1-dichloroethylene formed. The reaction mixture was then kept at room temperature for 2 h. The NMR spectrum of the carbon tetrachloride solution shows that 1,1-dichloroethylene was formed in 53 % of the theoretical yield (determined by using a reference solution of 1,1-dichloroethylene in CCl$_4$). 1,1-Dichloroethylene; IR (CCl$_4$) 1610 cm^{-1} (C=C); NMR (CCl$_4$) δ 5.5 ppm (s, H$_2$C=).

Decomposition of 2,2,2-trichloroethanesulfinyl chloride in excess base. To an aqueous sodium hydroxide solution (1 M, 200 ml) and carbon tetrachloride (5 ml) was slowly added 2,2,2-trichloroethanesulphinyl chloride (4.3 g, 0.02 mol). The reaction mixture was kept during 2 h at room temperature under nitrogen. Air must be excluded owing to the explosive nature of chloroacetylene in the presence of oxygen. The IR and NMR spectra of the carbon tetrachloride solution showed that chloroacetylene was formed together with 1,1-dichloroethylene. The yield of chloroacetylene was not determined. Chloroacetylene; IR (CCl$_4$) 3300 (CH) and 2100 cm^{-1} (C≡C); NMR (CCl$_4$) δ 1.78 ppm (s, CH). 1,1-Dichloroethylene was treated with a sodium hydroxide solution under the conditions described above. Chloroacetylene could not be detected in this experiment.

Bis(threo-2-chloro-1-methylpropyl)disulfide from cis-2-butene and sulfur monochloride.[6] cis-2-Butene (35 g) was added to a solution of FeCl$_3$ (1 g) in ethyl ether (150 ml). Sulfur monochloride (27 g, 0.2 mol) was then slowly added to the stirred solution over 30 min. cis-2-Butene was passed continuously through the reaction mixture to maintain an excess. The reaction mixture was stirred at room temperature for 3 h after addition of sulfur monochloride and then washed with water. The organic phase was dried and the solvent removed *in vacuo*. The residue was distilled under reduced pressure and a fraction was collected at b.p. 80−83 °C/0.2 mmHg; yield 19 g. Without further purification this fraction was used in the preparation of *threo*-2-chloro-1-methylpropanesulfinyl chloride (see below).

threo-2-Chloro-1-methylpropanesulfinyl chloride. A solution of bis(*threo*-2-chloro-1-methylpropyl)disulfide (18.5 g, 0.075 mol, a mixture of *meso* and *racemic* forms) and acetic anhydride (15.3 g, 0.15 mol) was cooled to 5 °C. Chlorine was passed into the solution at such a rate that the temperature remained between 10 and 15 °C. When no more chlorine was consumed, chlorine and acetyl chloride were removed by evaporation *in vacuo* at room temperature. The residue was distilled at reduced pressure to give *threo*-2-chloro-1-methylpropanesulfinyl chloride; b.p. 64−66 °C/2 mmHg; yield 15 g (57 %); n_D^{25} 1.5140; IR (film) 1150 cm^{-1} (S=O).

Decomposition of threo-2-chloro-1-methylpropanesulfinyl chloride in the presence of sodium hydroxide. *threo*-2-Chloro-1-methylpropanesulfinyl chloride (7 g, 0.04 mol) was added at room

temperature to an aqueous sodium hydroxide solution (2 M, 100 ml). The butene gas thus formed was passed through a sodium hydroxide solution to remove sulfur dioxide and then collected over water in a measuring cylinder. No more gas evolution could be observed after 2 h at room temperature. The gas (820 ml) was analysed at GLC (column, 4.5 m × 3 mm, packed with 15 % dimethylsulfolane on GasChrom RZ 60/80 mesh, relative retention times *trans*- and *cis*-2-butene 1.00:1.08, column temperature 30 °C). *cis*-2-Butene was present in more than 99 % purity.

Bis(erythro-2-chloro-1-methylpropyl)disulfide from trans-*2-butene and sulfur monochloride.*[6] This compound was first prepared according to the procedure described for the corresponding *threo*-isomer. However, because of the slow reaction of *trans*-2-butene and sulfur monochloride under these conditions a modified procedure was used. *trans*-2-butene (*ca.* 35 g) was added to a solution of chloroform (130 ml) and pyridine (0.2 ml). Sulfur monochloride (20.5 g, 0.152 mol) was then added slowly to the chloroform solution which was cooled with water. The reaction temperature was not allowed to rise above 25 °C. An excess of *trans*-2-butene was passed continuously through the reaction mixture. After the addition, the reaction mixture was kept at room temperature for 2 h. The chloroform and excess *trans*-2-butene were removed *in vacuo*. It was not possible to distil the residue at reduced pressure (0.2 mm) owing to decomposition. The crude residue was therefore used for the preparation of *erythro*-2-chloro-1-methylpropanesulfinyl chloride (see below).

erythro-2-Chloro-1-methylpropanesulfinyl chloride. A solution of crude bis(2-chloro-1-methylpropyl)disulfide (37.5 g, 0.152 mol), prepared as described above from *trans*-2-butene and sulfur monochloride, and acetic anhydride (31 g, 0.304 mol) was cooled to 5 °C. Chlorine was then passed into the solution at such a rate that the temperature remained between 10 and 15 °C. When no more chlorine was consumed the excess chlorine and acetyl chloride was evaporated *in vacuo* at room temperature. The residue could not be distilled owing to decomposition. Therefore, the crude *erythro*-2-chloro-1-methylpropanesulfinyl chloride was used for the decomposition experiment (see below) without further purification.

Decomposition of crude erythro-2-chloro-1-methylpropanesulfinyl chloride in the presence of sodium hydroxide. Crude *erythro*-2-chloro-1-methylpropanesulfinyl chloride (7 g) was decomposed as described above for the corresponding *threo*-isomer to yield *trans*-2-butene (280 ml) of more than 99 % purity.

Kinetic procedures. The rates of the decomposition of 2-chloroethanesulfinate have been measured at 21 °C and 34 °C in excess 0.5 M aqueous sodium hydroxide solution. The rates for the appearance of ethylene was followed

by collecting the gas in a measuring cylinder over water (21 °C).

Run a at 21 °C. 2-Chloroethanesulfinyl chloride (1.1178 g, 7.6 mmol) was added to a sodium hydroxide solution (0.5 M, 120 ml) to give 2-chloroethanesulfinate. The solution was kept at 21 °C and the gas was collected. Totally, 161 ml ethylene was evolved over 1396 min, which corresponds to an 85 % yield (correction was made for water pressure and atmosphere pressure). The rate of the decomposition was calculated from the slope of a plot of log $[a/(a-x)]$ *versus* time. (a = theoretical yield of ethylene; x = the ethylene gas evolved). The decomposition gave good first order kinetics up to 63 % reaction. The rate at 21 °C was $(16.1 \pm 1.5) \times 10^{-4}$ min^{-1}.

Run b at 34 °C. 2-Chloroethanesulfinyl chloride (1.1025 g, 7.5 mmol) was added to a sodium hydroxide solution (0.5 M, 120 ml). The solution was kept at 34 °C and the ethylene gas was collected. Totally, 171 ml ethylene was evolved over 225 min which corresponds to 90 % yield. The rate of the decomposition was derived as described in *run a.* The decomposition gave good first order kinetics up to 75 % reaction. The rate at 34 °C was $(14.7 \pm 0.7) \times 10^{-3}$ min^{-1}.

The activation energy 127.8 ± 8.4 kJ. mol^{-1} was obtained using the Arrhenius equation:

$$\log k_2/k_1 = (\Delta H_a/2.303R)[(T_2-T_1)/T_2 T_1]$$

where k is the rate constant, ΔH_a the activation energy, $R = 8.314$ J deg.$^{-1}$ mol^{-1} and T = absolute temperature.

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