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THE CATALYTIC PARTIAL OXIDATION OF ETHYL ALCOHOL IN THE VAPOR PHASE

THE USE OF A LIQUID SALT BATH FOR TEMPERATURE CONTROL

BY

DONALD B. KEYES

AND

WILLIAM LAWRENCE FAITH

BULLETIN No. 252

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THE USE OF A LIQUID SALT BATH FOR TEMPERATURE CONTROL

BY

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THE CATALYTIC PARTIAL OXIDATION OF ETHYL ALCOHOL IN THE VAPOR PHASE*

The Use of a Liquid Salt Bath for Temperature Control

I. Introduction

1. *Introductory.*—Since the discovery by H. D. Gibbs,† in 1916, of the catalytic oxidation of naphthalene to phthalic anhydride, vapor phase catalytic oxidation has become an important tool in the chemical industry. The theoretical possibility of making certain organic chemicals from very cheap raw materials by catalytic oxidation has led to a great deal of fruitful research in this field.

Among those reactions that have been studied quite extensively is the vapor phase catalytic oxidation of ethyl alcohol to produce acetic acid. However, the necessity of very close temperature control, in addition to other factors, has made progress in this direction difficult.

Temperature control is necessary because of the large amount of heat evolved by the reaction. At low temperatures, the oxidation is so slow that it is not commercially feasible, but at 350 deg. C. it becomes quite rapid, the temperature rises quickly, and the reaction is maintained at a high velocity by virtue of the heat liberated. If some of this heat is not taken away from the catalyst, the reaction will proceed beyond the formation of aldehyde and acid and give complete oxidation products (*i.e.,* carbon dioxide and water). Also, if the temperature of the catalyst is allowed to become too high, pyrogenic decomposition of the aldehyde takes place, with consequent diminution of yield.

In this bulletin an investigation of temperature control by means of a molten salt bath will be described.

2. *Acknowledgments.*—The investigation described in this bulletin was conducted by the Engineering Experiment Station of the University of Illinois, of which Dean M. S. Ketchum is the director, under the direction of Prof. D. B. Keyes, Head of the Division of Industrial Chemistry.

This investigation was made possible by the generous financial aid given by the Chemical Foundation, New York City, of which Mr. Francis P. Garvan is president, and Mr. W. W. Buffum general manager and treasurer.

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*This bulletin is the fifth publication of a series on this subject. The preceding numbers are: I, King, Swann and Keyes, J. Ind. Eng. Chem., 21, 1227, 1929; II, Keyes and Snow, ibid., 23, 561, 1931, and Univ. of Ill. Eng. Exp. Sta. Bul. 238, 1931; III, Faith and Keyes, J. Ind. Eng. Chem., 23, 1250, 1931; IV, Faith, Peters and Keyes, ibid., 24, 924, 1932.

†H. D. Gibbs, Can. Pat. 186,445, Sept. 10, 1918.
H. D. Gibbs, U.S. Pat. 1,284,887, 1918.

II. The Use of a Liquid Salt Bath for Temperature Control

3. *Historical.*—Attempts to modify or regulate the operating temperature of a catalyst in exothermic reactions have been limited almost exclusively to the chemical industry. Hence most of the results and conclusions derived therefrom have found their way into the patent literature only. This literature is crowded with designs of baths, agitators, and all manner of mechanical contrivances for regulating catalyst temperatures. Because of the unreliability and general chaotic character of the patent literature, any further discussion of its relation to temperature control will be avoided.

Only a few articles have been published in the scientific journals concerning this subject. In 1919 Huff* published an article, in the form of a review, in which he described general methods for control of exothermic catalytic oxidations.

Downs,† in 1926, described in detail a catalyst chamber for use in the catalytic oxidation of benzene, naphthalene, anthracene, and like compounds, which was basically a cleverly-designed mercury bath.

More recently, Faith and Keyes,‡ and Faith, Peters, and Keyes¶ have described various methods of temperature control of the catalyst in the vapor phase oxidation of alcohols.

4. *Method of Work.* — In previous articles by the authors,§** different methods of controlling the temperature of a catalyst in vapor phase partial oxidation of ethyl alcohol have been described. These methods have been mainly concerned with control of temperature by proper choice of the form of the catalyst, and variation of materials and construction of the catalytic chamber.

In the chemical industry, however, one of the preferred methods of temperature control of catalysts in exothermic processes is that in which the catalyst tube or chamber is immersed in a liquid bath. Since the temperature of this bath must be quite high, baths of mercury or molten metals, alloys, or salts are commonly used. Because of the widespread use of this means of temperature control it was thought advisable to compare one of these types with those methods which had already been tested in this laboratory.

A bath consisting of a eutectic mixture of $NaNO_3$ and KNO_3 was chosen as a typical bath. The eutectic salt mixture was chosen be-

*Huff, Trans. Am. Electrochem. Soc., 36, 167, 1919.
†Downs, J. Soc. Chem. Ind., 45, 188T, 1926.
‡Faith and Keyes, J. Ind. Eng. Chem., 23, 1250, 1931.
¶Faith, Peters, and Keyes, J. Ind. Eng. Chem., 24, 924, 1932.
§Faith and Keyes, J. Ind. Eng. Chem., 23, 1250, 1931.
**Faith, Peters, and Keyes, J. Ind. Eng. Chem., 24, 924, 1932.

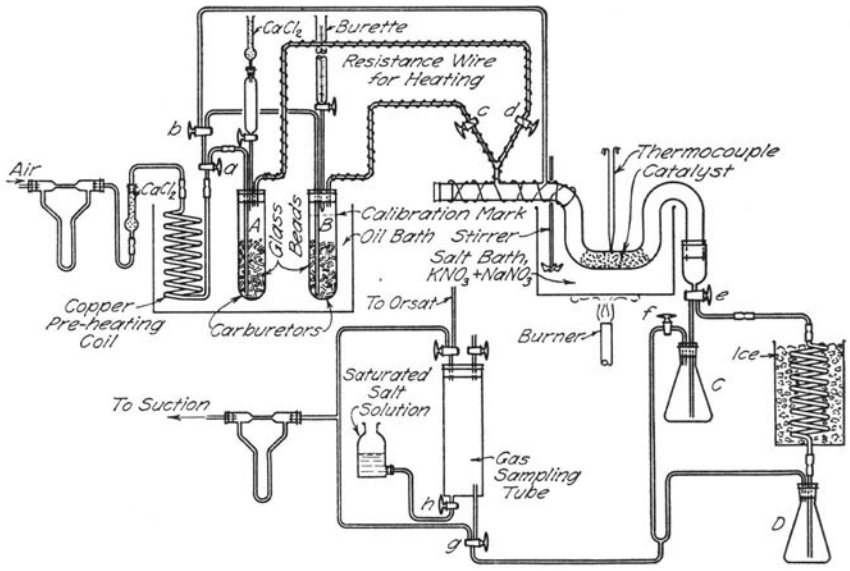

FIG. 1. DIAGRAM OF APPARATUS

cause of its low melting point (225 deg. C.), and because of the non-poisonous characteristics of its vapors.

The apparatus used consists essentially of that described formerly by Faith and Keyes,* with the exception that the furnace around the catalyst tube was replaced by the salt bath. A diagram of the apparatus used in this investigation is shown in Fig. 1. The Pyrex catalyst tube was bent in the shape of a U tube. The catalyst occupied the lower portion of the tube. The part of the U tube containing the catalyst was lowered into a welded steel box, about 6 inches on each side, filled with the molten salt mixture. The bath was heated by means of a large Meeker burner directly underneath, and, in order to facilitate quick heating, was insulated with asbestos board. The temperature of the bath was recorded by a chromel-alumel thermocouple and regulated by controlling the gas flow through the burner. A motor-driven stirrer kept the molten salt mixture at an even temperature throughout.

The operation of the apparatus was identical with that previously described. Air was passed through a flowmeter, where the rate of flow was measured, thence through a drying tower containing $CaCl_2$ to the preheater. The preheater was a copper coil immersed in the ther-

*Faith and Keyes, J. Ind. Eng. Chem., 23, 1250, 1931.

mostat. After passing through the preheater, the air was saturated with absolute ethyl alcohol in a carburetor also immersed in the thermostat. The air-alcohol vapors then passed through the catalyst tube in the salt bath, where the alcohol was oxidized to acetaldehyde, acetic acid, and carbon dioxide. The liquid products and unchanged alcohol were condensed and collected in a receiver; the gaseous products and some of the aldehyde formed passed through a sampling tube and flowmeter to the atmosphere.

5. *Analysis of Products.*—

(a) Determination of Acetaldehyde

The aldehyde in the condensate was determined by the neutral sodium sulphite method.*

(b) Determination of Acetic Acid

The acid in the condensate was titrated with N sodium hydroxide solution. The acid was identified by the method of DuClaux, as described by Kamm.†

(c) Determination of Unchanged Ethyl Alcohol

The unchanged ethyl alcohol was determined by the ammoniacal silver nitrate method described by Lowdermilk and Day.‡

(d) Orsat Analysis

The gas sample was delivered from the sampling tube to the Orsat and then passed through 30-per-cent sodium acid sulphite solution to determine aldehyde. Carbon dioxide was determined by passing the residual gas through 30-per-cent potassium hydroxide solution.

III. Discussion of Results

6. *Results.*—The results obtained using the salt bath as a method of temperature control are shown in the tables. Table 1 shows the results obtained at high alcohol-air ratios; *i.e.*, those which are conducive to high yields of intermediate products. It is quite noticeable that at the alcohol-air ratios above unity the oxidation really becomes a dehydrogenation process due to an excess of alcohol. These results are much like those obtained using a plain Pyrex tube with a removable furnace as a means of temperature control. However,

*Kingscott and Knight, "Methods of Quantitative Organic Analysis," p. 245, Longmans, 1914.
†Kamm, "Qualitative Organic Analysis," p. 139. Wiley, 1922.
‡Lowdermilk and Day, J. Am. Chem. Soc. 52, 3535, 1930.

TABLE 1

RESULTS USING PYREX TUBE CATALYST CHAMBER IN LIQUID SALT BATH

Copper turnings catalyst
Length of catalyst, 45 mm.; diameter of catalyst, 13 mm.

Thermostat Temperature deg. C.	Alcohol-Air Ratio, g. EtOH per liter air	Space Velocity* (cc. gas per cc. catalyst per min.)	Flow, liters per min.	Temperature Catalyst Bath deg. C.	Conversions				Yield
					Aldehyde per cent	Acid per cent	CO_2 per cent	Total per cent	per cent
54	0.67	119	0.54	415	55.8	0.8	6.8	64.1	89.5
57	0.80	118	0.51	415	54.5	0.6	4.9	60.0	91.7
60	1.01	122	0.495	350	55.5	1.2	0.9	57.6	98.4
60	1.01	122	0.495	410	56.8	0.9	1.5	59.2	97.4
60	1.01	122	0.495	440	55.4	0.9	1.5	57.8	97.4
63.5	1.33	131	0.48	350	46.5	0.9	0.5	47.9	99.0

*The space velocities recorded in this bulletin are calculated on the basis of gas leaving the carburetor referred to standard conditions of temperature and pressure.

TABLE 2

RESULTS USING COPPER-GLASS SEAL CATALYST CHAMBER

Copper turnings catalyst
Length of catalyst, 45 mm.; diameter of catalyst, 13 mm.

Thermostat Temperature deg. C.	Alcohol-Air Ratio, g. EtOH per liter air	Space Velocity (cc. gas per cc. catalyst per min.)	Flow, liters per min.	Temperature Catalyst Bath deg. C.	Conversions				Yield
					Aldehyde per cent	Acid per cent	CO_2 per cent	Total per cent	per cent
45.0	0.38	120	0.61	480	49.6	6.9	24.6	81.1	69.9
53.0	0.59	115	0.54	435	54.9	7.4	13.0	75.3	82.7
54.0	0.62	122	0.57	415	55.4	6.8	7.7	69.9	88.9
57.0	0.77	116	0.51	415	56.8	4.1	3.5	64.4	94.5
61.0	0.94	116	0.48	410	54.7	4.4	1.6	60.7	97.3
Same as above except Pyrex tube									
54.0	0.66	125	0.57	415	56.0	2.4	6.0	64.4	90.6
57.0	0.77	116	0.51	415	53.9	1.8	4.1	59.8	93.1
61.0	0.97	124	0.51	410	52.7	1.3	2.1	56.1	96.3
61.0	0.94	148	0.61	420	45.6	1.5	2.2	49.3	95.6

they are inferior to those obtained using a copper tube sealed to glass leads, since the acid conversion is much higher in the latter case.

The two types of catalyst tubes mentioned are those which were found to be superior to others in former studies by the authors. Typical results obtained with these tubes are shown in Tables 2 and 3, for the sake of comparison.

Tables 4 and 5 show the results obtained at an alcohol-air ratio of about 0.4 g. alcohol per liter of air. This alcohol-air ratio is that which has been found favorable to high aldehyde conversions.

TABLE 3

RESULTS USING PYREX TUBE CATALYST CHAMBER

Copper turnings catalyst
Length of catalyst, 45 mm.; diameter of catalyst, 13 mm.
Thermostat temperature, 45 deg. C.

Flow, liters per min.	Alcohol-Air Ratio, g. EtOH per liter air	Space Velocity (cc. gas per cc. catalyst per min.)	Temperature deg. C.	Conversions				Yield
				Aldehyde per cent	Acid per cent	CO_2 per cent	Total per cent	per cent
0.51	0.38	100	462	71.0	3.1	12.0	86.1	86.1
0.52	0.38	103	465	72.0	3.5	12.0	87.5	86.3
0.55	0.38	108	470	73.0	3.3	11.6	87.9	86.7
0.60	0.38	118	488	76.0	4.6	12.4	93.0	86.6
0.62	0.37	121	512	78.0	2.8	14.1	94.9	85.1
0.64	0.36	123	517	77.0	2.6	13.5	93.1	85.5
0.65	0.36	126	520	76.5	2.2	10.9	89.6	87.8
0.66	0.35	128	522	76.0	2.0	11.1	89.1	87.5
0.74	0.31	141	542	74.0	1.3	11.4	86.7	86.8

TABLE 4

RESULTS USING PYREX TUBE CATALYST CHAMBER IN LIQUID SALT BATH

Copper turnings catalyst
Length of catalyst, 45 mm.; diameter of catalyst, 13 mm.
Thermostat temperature, 45 deg. C. Flow, 0.51 liters per min.
Alcohol-air ratio, 0.39 g. EtOH per liter air.
Space velocity, 101 cc. gas per cc. catalyst per min.

Temperature Catalyst Bath deg. C.	Conversions				Yield
	Aldehyde per cent	Acid per cent	CO_2 per cent	Total per cent	per cent
300	16.1	3.7	0.7	20.5	96.6
355	57.7	5.2	22.1	85.0	74.0
400	62.3	1.0	24.7	88.0	71.9
420	63.3	1.2	21.4	85.9	73.9
440	61.2	0.9	21.4	83.5	74.4
460	59.1	0.7	20.3	80.1	74.6
470	59.8	0.8	20.3	80.9	74.9
483	61.6	1.1	20.0	82.7	75.8
487	59.9	0.7	21.9	82.5	73.4

The results obtained using the salt bath around the catalyst tube are in between those found using a plain Pyrex tube in air and a copper tube sealed to glass leads.

The aldehyde conversions approach those obtained by use of the Pyrex tube chamber in air, but the carbon dioxide conversions are much higher. In fact, the conversions to carbon dioxide are almost identical with those obtained using the copper-glass seal tube.

It is quite probable that the high carbon dioxide conversions are due to the excessive time of heating of the entrance and exit tubes to

TABLE 5

RESULTS USING PYREX TUBE CATALYST CHAMBER IN LIQUID SALT BATH

Copper turnings catalyst
Length of catalyst, 45 mm.; diameter of catalyst, 13 mm.
Thermostat temperature, 46 deg. C. Flow, 0.61 liters per min.
Alcohol-air ratio, 0.405 g. EtOH per liter air.
Space velocity, 121 cc. gas per cc. catalyst per min.

Temperature Catalyst Bath deg. C.	Conversions				Yield
	Aldehyde per cent	Acid per cent	CO_2 per cent	Total per cent	per cent
310	22.3	0.3	0.8	23.4	96.6
350	61.3	3.4	21.3	86.0	75.2
425	60.7	1.2	19.4	81.3	76.1
445	63.2	0.7	20.6	84.5	75.6
460	61.9	1.4	20.6	83.9	75.4
485	69.3	1.6	22.2	93.1	76.2
500	67.3	1.2	19.9	88.4	77.5

the catalyst chamber, on account of the large amount of heat in the neighborhood of the salt bath.

Also, it is quite probable that the measured temperature of the salt bath is not the same as that of the catalyst itself. The tables show that the reaction becomes sufficiently fast at a temperature of 350 deg. C. to maintain itself by its exothermic character. Consequently, the heat given off by the reaction cannot be taken away from the catalyst by a bath at 350 deg. C. as easily as it can be radiated from a hot tube to the surrounding air, as was the case when only a plain tube in air was used.

Hence at temperatures above 350 deg. C. (and the reaction goes only slowly below that temperature) the salt bath is a hindrance rather than a help, because it lowers the temperature gradient from the catalyst to the surroundings, and thus actually helps the temperature of the catalyst to build up.

The reverse was true, of course, when the copper-glass seal tube was used. The copper conducted the heat away from the catalyst so fast that it could not maintain the reaction itself, and thus required a constant heat input.

7. *Interpretation of Results.*—It can be said that the salt bath as a means of temperature control in vapor phase catalytic partial oxidation of ethyl alcohol is inferior to other methods described by the authors previously.

This is not because the temperature of the salt bath cannot be maintained at a constant temperature (for this is its predominant feature), but because it lowers the temperature gradient from catalyst

to surroundings and helps build up excessive temperature in the catalyst chamber.

If it is desired to oxidize ethyl alcohol and obtain a high conversion of acetaldehyde in a single pass, then the use of the plain Pyrex catalyst tube, in which the reaction is maintained by its exothermic character, is by far the best method that has been studied. The copper-glass seal chamber, which requires intermittent heating, yields very poor results in this respect, giving low aldehyde and high carbon dioxide conversions.

This may be seen by comparison of Tables 2 and 3 with Tables 4 and 5.

If a high acid yield is the desired objective of the reaction, the copper-glass seal tube is the preferred means, and the salt bath control method falls in between this method and the one in which the Pyrex tube chamber in air is used.

It should be noticed that the maximum acid conversion for the salt bath method appears at a much lower temperature than in the other cases. However, this recorded temperature is the temperature of the salt bath and not necessarily that of the catalyst, so it is possible that the temperatures of maximum conversion are not so far apart.

IV. Conclusions

8. *Summary and Conclusions.*—A study has been made of the use of a salt bath as a means of temperature control for the catalytic vapor phase partial oxidation of ethyl alcohol.

The results obtained using the salt bath as a method for temperature control have been found to fall between those obtained with the plain Pyrex tube catalyst chamber and the copper-glass seal chamber.

When a high single pass conversion to acetaldehyde is desired, the plain Pyrex tube method is best. The salt bath method is next best in this respect, and the copper-glass seal chamber is the poorest.

The highest conversion to acid has been obtained by using the copper-glass seal chamber. In this capacity, the salt bath is better than the plain Pyrex tube.

The inefficiency of the salt bath as a means for temperature control of catalytic oxidations is probably due to the low temperature gradient between the catalyst and the bath, thus rendering it difficult for heat to leave the catalyst.

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