



AN INDIRECT AROCLOR HEATER for UNIT CHEMICAL OPERATIONS

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Aroclor* 1248 (chlorinated biphenyl) is an ideal nonflammable liquid phase heat-transfer medium for temperatures up to 300°C.

This bulletin describes the physical properties of Aroclor 1248 and illustrates the design and operation of heaters that have been used successfully by Monsanto plants during the past seven years. The units described are gas fired and the capacities are in the range of 200,000 to 400,000 B.t.u. per hour.

Other larger commercial installations using Aroclor 1248 have capacities ranging up to 2,000,000 B.t.u. per hour.

Also small electrically heated stationary and portable units with capacities around 40,000 B.t.u. per hour, using Aroclor 1248 as the heat-transfer medium, are in service.

Several leading manufacturers of heating equipment are in position to build or offer Aroclor heating units to meet specific requirements.

*Reg. U. S. Pat. Office

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UNIT CHEMICAL OPERATIONS

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An Indirect Aroclor Heater for Unit Chemical Operations

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The characteristics of Aroclor 1248 indicate that it is an ideal liquid³ phase heat-exchange medium for temperatures up to 300° C. This article discusses its properties and illustrates the design and operation of heaters that have been used successfully by Monsanto plants during the past seven years.

IN CERTAIN manufacturing processes of the Monsanto Chemical Company, it was necessary to employ a noncombustible heat-transfer medium at pressures of 80 pounds per square inch or less and temperatures up to 300° C. The following general properties of a heat-exchange medium were required:

- Freedom from fire hazards.
- Viscosities to permit pumping at room temperatures.
- Boiling point sufficiently above 300° C. to assure a liquid condition at all times.
- Stability against heat, with enough safety factor to accommodate accidental overheating.
- Controllable vaporization losses.
- Freedom from corrosive action against valves, piping, tank jackets, etc., made of cast iron and steels, bronze, and stainless steel.
- Freedom from toxicity hazard.

Aroclor, chlorinated biphenyl (registered in U. S. Patent Office), was selected. The pertinent physical characteristics relative to its use as a heat-transfer medium are given in Table I.

FREEDOM FROM FIRE HAZARDS

FLASH POINT. Limitations of this test for the prediction of the fire hazard of relatively nonvolatile organic fluids have been recognized by the American Society for Testing Materials committee (3, 4).

FIRE POINT is a more significant measurement. The Underwriters' Laboratories (10) state that fire tests more truly reflect the (lack of) fire hazard of Aroclor.

SPONTANEOUS IGNITION TEMPERATURE. The combustion-resisting qualities of Aroclor 1248 are indicated by its high spontaneous ignition temperature of 704° C. (1299° F.) determined by Sullivan, Wolfe, and Zisman (9), using the convenient apparatus described by Sortman, Beatty, and Heron (8).

Under conditions of industrial use the spontaneous ignition temperature will be determined by factors including the nature of the hot surface, the amount of liquid impinging on it, the volume of enclosed space, and the ventilation.

An accidental failure in a heating system demonstrated the nonflammability of Aroclor 1248 and its freedom from the hazard of fire propagation. An operator's failure to start the circulation of the heat-transfer medium when the gas heater was on resulted in excessive coil temperatures and caused the lower coil to soften and sag into the fire chamber. A weld ruptured and Aroclor 1248 poured into the red-hot fire chamber in contact with the flame. Dense smoke arose from the heater but there was no external fire. After the gas flame was cut off, the smoking stopped.

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SPRAY FLAMMABILITY. When a tube ruptures in a liquid heat-transfer system under high pressure, a spray or mist forms. The possible fire hazard under these conditions requires considerations not covered by the foregoing discussions relative to the material in the liquid form. In their comprehensive study of flammability of the higher boiling liquids and their mists, Sullivan, Wolfe, and Zisman (9) determined the spray flammability limit of numerous materials in accordance with the percentage of oxygen required for combustion. This value was then correlated with the results of incendiary firing tests of the fluids conducted at the Naval Proving Ground, Dahlgren, Va. The oxygen requirement for Aroclor 1248 combustion in the spray flammability limit studies was found to be 64%. Fluids requiring over 45 to 50% oxygen in the spray test failed to cause a fire in the incendiary test. These results establish the nonflammable and noncombustible qualities of Aroclor 1248.

VISCOSITY CONSIDERATIONS

COLD FLOW. For most inside installations centrifugal pumps will handle Aroclor at reduced rates without preheating. Experience confirms this. Outside installations have been started at 0° C. by heating the pump and feed line until circulation through

TABLE I. PHYSICAL PROPERTIES OF AROCLOR 1248

Appearance	Practically colorless mobile liquid	
	° C. (° F.)	
Absolute density, g./ml.	1.44	30 (86)
	1.41	60 (140)
	1.37	100 (212)
	1.32	200 (392)
	1.17	300 (572)
Absolute viscosity, centipoises	112	30 (86)
	17.5	60 (140)
	4.2	100 (212)
	0.69	200 (392)
	0.47	300 (572)
Thermal conductivity, B.t.u./hour/sq. foot/° F./foot	0.0613	30 (86)
	0.0688	60 (140)
	0.0860	100 (212)
Distillation range, A.S.T.M. D-20, ° C.	340-375	
Flash point, Cleveland open cup, A.S.T.M. D-92-45, ° C.	193-196	
Fire point, Cleveland open cup, A.S.T.M. D-92-45	None	
Pour point, A.S.T.M. D-7, ° C.	-7	
Coefficient of expansion, ml./ml./° C. (25-65° C.)	0.000702	
Specific volume, ml./g.	0.596	30 (86)
	0.709	60 (140)
	0.728	100 (212)
	0.787	200 (392)
	0.860	300 (572)
Specific heat, cal./g./° C.	0.383	50 (122)
	0.287	100 (212)
	0.228	200 (392)
	0.355	300 (572)
Vapor pressure, mm. Hg	0.00037	37.8 (100)
	0.16	100 (212)
	2.3	180 (356)
	18.0	200 (392)
	360.0	300 (572)

TABLE II. STABILITY OF AROCLOR 1248 HEATED FOR 30 HOURS

Temperature, °C.	Mg. of HCl per Gram of Aroclor
280	0.079
300	0.186
310	0.199
320	0.222
330	0.248

TABLE III. STABILITY OF AROCLOR 1248 CONTINUOUSLY HEATED AT 280° AND 330° C.

Hours	Mg. of HCl per Gram of Aroclor	
	280° C.	330° C.
30	0.076	0.248
60	0.116	0.310
90	0.159	0.425
120	0.194	1.141
150	0.261	1.802

TABLE IV. GAS ANALYSIS OF AROCLOR 1248 HEATED 4 HOURS AT 260° C. AND 210 POUNDS PER SQUARE INCH PRESSURE

Gas	%
Carbon dioxide	None
Carbon monoxide	None
Oxygen (derived from air)	20.2
Methane	0.2
Hydrogen chloride	None
Chlorine	None
Acidity (% by weight HCl)	
Prior to exposure	0.0075
After exposure	0.0075

the heater had been effected so that the system could operate normally.

BOILING POINT

Operating experience has shown that the boiling point of Aroclor 1248 (340° C. at 760 mm.) is enough above the 300° C. operating limit to prevent trouble from this cause. At the maximum operating temperature the vapor pressure is less than 0.5 atmosphere.

HEAT STABILITY

In order to establish a maximum practical operating temperature, the stability of the material when heated to elevated temperatures in the presence of iron was noted. A slow stream of nitrogen was passed over the hot Aroclor to sweep the decomposition products into a caustic trap. The amount of acidic material was determined and calculated as hydrochloric acid. The results given in Table II indicate the stability of Aroclor 1248 when individual samples were heated for 30 hours at the given temperatures. The decomposition is very low at temperatures up to 300° C.

The stability of Aroclor 1248 in contact with iron continuously heated at 280° and 330° C. is indicated by Table III. These test results indicate that Aroclor 1248 in contact with iron can be used satisfactorily at temperatures up to 300° C.

The National Board of Fire Underwriters (10) reported that "decomposition of the product [Aroclor 1248] was not appreciable at temperatures below 400° C., but became increasingly apparent at higher temperatures."

Their workers analyzed the decomposition products of Aroclor 1248 heated in the presence of hot iron at 459° C. and also heated in an iron cylinder

at 340° C. fitted with an internal gas burner so that the gas flame impinged directly on the surface of the Aroclor. Analysis of the gases produced under these conditions included 0.6% carbon monoxide, 0.17% oxygen (derived from the air), 0.99% hydrogen chloride, 2.1% combustible gas calculated as methane, 0.002% chlorine, and no phosphene.

The same workers studied the stability of Aroclor when heated for 4 hours in an iron pipe at 280° C. under an internal pressure of 210 pounds per square inch, resulting from the introduction of compressed air. Following this treatment and cooling, the gases removed from the system were analyzed. The acidity of the Aroclor was determined prior to and after exposure to these conditions.

The results given in Table IV reflect an extremely small amount of decomposition under conditions similar to those selected for the practical use of Aroclor 1248 as a heat-transfer medium.

In actual practice using the type of heating unit described below there has been no evidence of hydrochloric acid effect and no carbon deposits have been noted in the heater, pipes, or valves. The heaters have been operated successfully with combustion gases at approximately 600° C. in contact with the coils.

At one time, a spiral coil type of heater was built and through faulty design a burner tunnel was located 1.5 inches from the 2-inch steel pipe coil. The radiation from the white-hot refractory tunnel and the direct impingement of the flame on the pipe caused the pipe to glow a dark red for a space of about 2 inches. This unit was operated almost 3 months before a circulation stoppage occurred. When the line was opened lumps of carbon were found to have come loose and moved forward to lodge in the globe valves. The lumps measured about 1 × 2.5 cm. and one face was formed to fit the pipe curvature. No hydrochloric acid effect was noted and the system was put back into operation after the burner location was changed.

CONTROLLABLE VAPORIZATION LOSSES

The heating systems in which Aroclor 1248 is used are sealed except for the expansion tank, which has a loose cover or a vent pipe. Because this tank is separated from the stream by a substantial length of pipe, the temperature in the tank remains low.

In the light of the relatively low vapor pressure values for Aroclor 1248 (7), and as it is unlikely that the expansion tank will ever reach even the 1-mm. state (130° C.), there should not be a measurable loss of Aroclor from the system. Actual experience bears out this contention.

OTHER LOSSES. Although Aroclor darkens on use, its characteristics remain the same and replacement has not been found necessary after 7 years of continuous use. Accidental leaks or spills constitute the only observed losses.

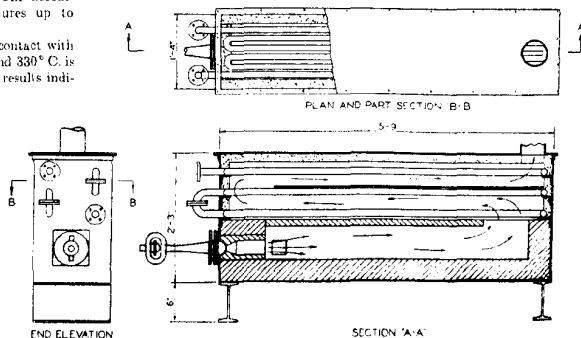


Figure 1. Heater

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TABLE V. RESISTANCE OF STRUCTURAL MATERIALS TO AROCLOR 1248

Metals	At 25° C.	At 125° C.	Metals	At 25° C.	At 125° C.
Aluminum	R	R	Zinc	R	R
Copper	R	R	Mild steel	R	R
Magnesium	R	R	Phosphor bronze	R	D
Nickel	RH	R	Red brass	D	D
Silver	R	R	Stainless steel	RH	RH
Tin	R	R	Yellow brass	R	R

RH. Excellent resistance, less than 1.0×10^{-4} cm. per day penetration or 0.00014 inch per year.

R. Good resistance, penetration between 1.0×10^{-4} and 10×10^{-4} cm. per day or between 0.00014 and 0.0014 inch per year.

D. Doubtful resistance, penetration between 10×10^{-4} cm. per day and 100×10^{-4} cm. per day or between 0.0014 and 0.014 inch per year.

e. Following letter indicating resistance, signifies material may be better than indicated if totally immersed, as weight loss is believed to come from oxidation of part of test strip exposed to air.

FREEDOM FROM CORROSIVE ACTION

The resistance of various metals (7) at 25° and 125° C. is given in Table V.

Similar studies made at 325° C. (6) indicate that the penetration, in inches per year, for mild steel is 0.0028; for yellow brass, 0.0047; for copper, 0.0015.

Many years of practical operating experience with Aroclor 1248 as a heat-transfer medium have shown that the material is practically noncorrosive to valve, piping, tank jackets, etc., made of cast iron and steel, bronze, and stainless steel.

FREEDOM FROM TOXICITY HAZARD

Aroclor 1248 is a very stable, unreactive liquid. If the material is spilled on the skin, there are no noticeable ill effects; however, it is well to wash the skin with soap and water after contact.

A skin burn resulting from accidental contact with hot Aroclor should be treated in the normal procedure used for hot oil burns. Aroclor adhering to the burned area need not be removed immediately unless treatment of the burn demands it; in this case soap and water or repeated washings with a vegetable oil (flinseed oil) should be used.

The vapors emitted by Aroclor 1248 heated to elevated temperatures are injurious to the liver on prolonged exposure and should not be breathed. Drinker (5) indicated that 0.5 mg. of Aroclor 1248 per cubic meter of air is the maximum safe amount permissible in workrooms.

In commercial heat-transfer installation, the presumption is that the Aroclor is in a closed system free from leaks. Accordingly, there should be no opportunity for workers to come in contact with vapors from the hot heat-transfer medium.

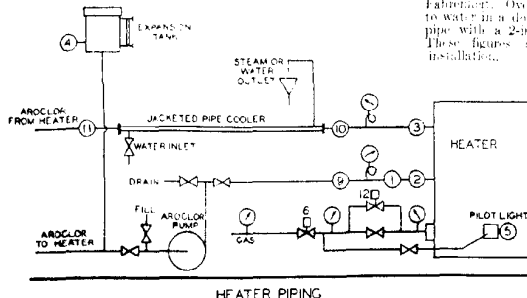


Figure 2. Cooler, Pump, and Expansion Tank

DESIGN AND CONSTRUCTION

HEATING SYSTEM. The heating system required to utilize Aroclor 1248 as the exchange medium includes a heater (Figure 1), cooler, pump, and expansion tank (Figure 2), and control and safety circuits (Figure 3).

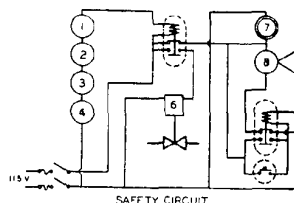


Figure 3. Control and Safety Circuit

HEATER DESIGN. The general requirements of the heater are compactness, ease of construction and service, and avoidance of direct flame impingement on the tubes.

Direct flame impingement is prevented by directing the gas flame through an enclosed channel with half-thickness fire brick protecting the tubes above until the high temperature of the flame is reduced by heat radiated through these bricks and reflected to the two bottom coils. The size of the heater is reduced to a minimum by using only 4.5 inches of insulating fire brick in the hottest areas and 2 inches of Eagle No. 66 insulation on the cooler spots.

Two sizes of units are used. One has a maximum capacity of 200,000 B.t.u. per hour, as shown in Figure 1. The larger has a range of 20,000 to 100,000 B.t.u.

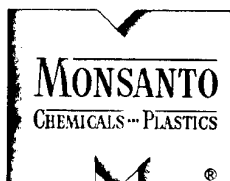
The small size consists of 90 feet of 1-inch standard weight steel pipe made into three coils of six pipes each and connected in series. The larger size consists of 144 feet of 1.5-inch standard weight steel pipe arranged in the same fashion. Results of tests on the small-size heater covering temperatures from 150° to 300° C. show temperature rises to 10° to 20° C. in the Aroclor while circulating at a rate of 15 to 20 gallons per minute. The outlet gas temperature is consistently within 75° C. of the outlet Aroclor temperature. It is observed that the bottom two coils absorb the major part of the heat. The small unit requires 20 to 60% efficiencies for capacities up to 200,000 B.t.u. per hour when a natural gas of 1000 B.t.u. per cubic foot is burned. A Surface Combustion Company high-pressure inspirator and tunnel burner set is used in this installation.

Overall heat-exchange coefficients (U) for the furnace are in the range of 7 to 10 B.t.u. per hour per square foot per degree Fahrenheit. Overall heat-exchange coefficients for Aroclor 1248 to water in a double pipe heat-exchanger made from 1.25-inch pipe with a 2-inch pipe jacket are in the 120 to 180 range. These figures are calculated from test results on this installation.

COOLER. A very useful adjunct to the system is a double pipe cooler on the outlet of the heater shown in Figure 2. By running water into the jacket, the Aroclor can be cooled and the temperature of the system lowered. This is helpful when it is necessary to cool a batch before removing it or when an exothermic reaction starts to get out of control.

PUMP. The pump for circulating the Aroclor 1248 may be any one of a number of standard centrifugal units designed for hot liquid service.

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