Liquid−Liquid Equilibrium of (Lube-Oil Cut + Furfural) in Several Solvent/Feed Ratios and at Different Temperatures

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Experimental data for liquid−liquid equilibrium of the lube-oil cut + furfural system is determined. The effect of the solvent-to-feed ratio and the operating temperature on the distribution of aromatic, naphthenic, paraffinic, and furfural in extract and raffinate phases is investigated. We determined the aromatic, naphthenic, and paraffinic content of raffinate and extract phases by using the ASTM standard methods.

Introduction

Today, by increasing the number and capacity of industrial plants, demand for lubricating oils has been increased to decrease the undesirable damages caused by friction.[1] Lubricating oils are one of the main and useful groups of petroleum products. In the process of producing lubricating oils, aromatic hydrocarbons should be removed from lube-oil cuts to develop various lubricating properties.[2−4] Lube-oil cuts normally contain paraffinic, aromatic, and naphthenic components that are generally separated from one of the heavy petroleum fractions of the vacuum distillation column under specific operational conditions. Liquid−liquid extraction is the most common method for separation of aromatics from lube-oil cuts. These aromatics should be reduced by a selective and suitable solvent that maximizes the differences in properties between the liquid phases.[3,5−7] Furfural is one of the solvents with the most wide usage that is selective enough toward aromatics. By increasing the temperature, the selectivity of furfural decreases, and it is acceptable for light and heavy vacuum distillates, as reported by many authors.[8−11] To remove aromatic hydrocarbons, furfural and lube-oil cut are normally mixed together in a mixer-settler. After suitable agitation and appropriate settling time, two immiscible phases are produced. The light phase, which is enriched with linear and cyclic saturated hydrocarbons (paraffinic and naphthenic), is called raffinate, which is used as feed in the lubricating oil production process. The heavy phase is called extract, which contains the furfural and aromatic compounds. To separate furfural from other components, the extract phase should be sent to a solvent recovery unit. The design and simulation of solvent recovery processes is normally performed by empirical methods that require expensive experimental information.[12] Also, several methods have been proposed for recycling used lubricating oils and solvent extraction byproducts.[13−15]

Empirical methods are used to define the composition of complex hydrocarbon mixtures such as petroleum fractions because it is not possible to identify all of the individual components present.[16] The pseudocomponents approach based on distillation curves[17] used in vapor−liquid equilibrium calculations is not useful for liquid−liquid extraction (LLE) because the chemical structure has a much larger effect on LLE compared with boiling temperature.[6,7] For the determination of the composition of complex mixtures, standard test methods such as ASTM D2007[18] can be used. Because these methods are time and money consuming, the composition of petroleum mixtures is often determined by the use of their physical properties.[6] Riazi and Daubert[19] have proposed relations to calculate the composition of aromatic, naphthenic, and paraffinic compounds from the viscosity and refractive index of the mixture. They also reported the main parameters of characterization for petroleum fractions.[20] Peng and Robinson proposed a simple procedure for characterizing the heavy petroleum fractions.[21] Coto et al.[6,7,11] used specific gravity (SG), refractive index (RI), density (*D*), and sulfur content to determine the composition of complex hydrocarbon mixtures. They considered the liquid phases (feed, raffinates, and extracts) formed by three groups of pseudocomponents: saturates (S), aromatics (A), and polar (P), following the ASTM D2007 standard test method.

The solvent-to-feed ratio and operational temperature are the main effective parameters on the separation of aromatics. To show the effect of these parameters, the liquid−liquid equilibrium is used. Marigliano et al.[23] studied the influence of temperature on the liquid−liquid equilibrium in a ternary system. This work focuses on the determination of liquid−liquid equilibrium of this system at different temperatures and several solvent/lube-cut ratios.

Table 1. Physical Properties of Lube-Oil Cut and Furfural

	furfural	lube-oil cut
ρ (20 °C)/g·cm^{-3}	1.1598	0.9257
η (25 °C)/pa·s	1.49	445
t_f/°C	61.7	258.3
MW	96	467
n (20 °C)	1.5261	1.5208
100w_A		25.1
100w_N		12.7
100w_P		62.2

Materials and Methods

The lube cut of Isfahan refinery was used for extraction of lube oil using furfural. The physical properties of lube-oil cut and furfural are shown in Table 1. w_A, w_N, and w_P are mass fractions of aromatic, naphthenic, and paraffinic hydrocarbons, respectively.

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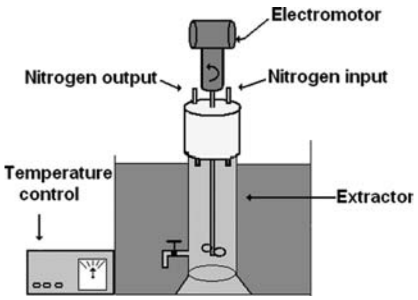

Figure 1. Experimental setup used in extraction operation.

Table 2. Properties of Extract Phase for Furfural (1) + Lube-Oil Cut (2) System at Several Temperatures

		n	ρ (20 °C)		furfural		
t/°C	(V_1/V_2)	(20 °C)	g·cm^{-3}	$100w_S$	($100w_1$)	$100w_A$	$100w_N$
50	7.5	1.57871	1.0353	4.53	95.24	40.47	23.94
50	6	1.58256	1.0388	4.65	92.77	43.51	20.66
50	4.5	1.58552	1.0421	4.73	90.12	45.25	18.89
50	4	1.58886	1.0473	4.78	88.78	46.56	18.51
50	3	1.59312	1.0512	4.95	85.86	49.31	16.09
50	2	1.59623	1.0552	5.11	82.71	51.36	13.63
50	0.75	1.59937	1.0594	5.28	79.32	53.13	11.98
50	0.375	1.60389	1.0633	5.33	75.46	56.24	10.53
60	7.5	1.56976	1.0197	4.32	93.46	37.52	22.88
60	6	1.57268	1.0215	4.45	90.83	40.37	20.15
60	4.5	1.57531	1.0238	4.63	88.18	42.13	18.12
60	4	1.57689	1.0251	4.66	86.8	43.24	17.82
60	3	1.58207	1.0314	4.84	83.76	45.94	15.15
60	2	1.58603	1.0371	5.08	80.61	48.17	12.97
60	0.75	1.58894	1.0409	5.22	77.05	49.87	11.47
60	0.375	1.59352	1.0467	5.28	73.13	52.42	11.13
70	7.5	1.55981	1.0032	4.05	90.56	33.93	22.48
70	6	1.56235	1.0041	4.12	88.07	36.71	19.54
70	4.5	1.56517	1.0073	4.42	85.38	38.43	17.03
70	4	1.56672	1.0087	4.48	83.93	39.26	16.85
70	3	1.57154	1.0144	4.74	80.81	41.89	14.52
70	2	1.57567	1.0202	4.95	77.72	44.37	12.44
70	0.75	1.57842	1.0238	5.21	74.25	45.93	11.02
70	0.375	1.58288	1.0293	5.28	70.42	48.32	10.22
80	7.5	1.54973	0.9868	3.88	89.24	30.22	23.22
80	6	1.55306	0.9887	4.03	86.73	33.17	19.83
80	4.5	1.55625	0.9923	4.26	84.02	35.25	17.61
80	4	1.55806	0.9936	4.29	82.59	36.37	17.43
80	3	1.56191	0.9988	4.58	79.46	38.12	15.44
80	2	1.56672	1.0046	4.78	76.22	41.17	12.03
80	0.75	1.57049	1.0098	5.05	72.63	43.29	10.11
80	0.375	1.57327	1.0124	5.11	68.65	45.11	9.86
90	7.5	1.54396	0.9768	3.79	87.45	28.24	21.98
90	6	1.54723	0.9796	3.94	84.84	30.88	19.12
90	4.5	1.55061	0.9831	4.16	82.21	32.94	17.31
90	4	1.55272	0.9848	4.22	80.89	34.31	16.75
90	3	1.55736	0.9909	4.44	77.74	36.52	14.75
90	2	1.56228	0.9967	4.61	74.53	39.77	11.60
90	0.75	1.56554	1.0013	4.87	71.05	41.54	10.62
90	0.375	1.57057	1.0081	5.02	67.24	43.95	9.46

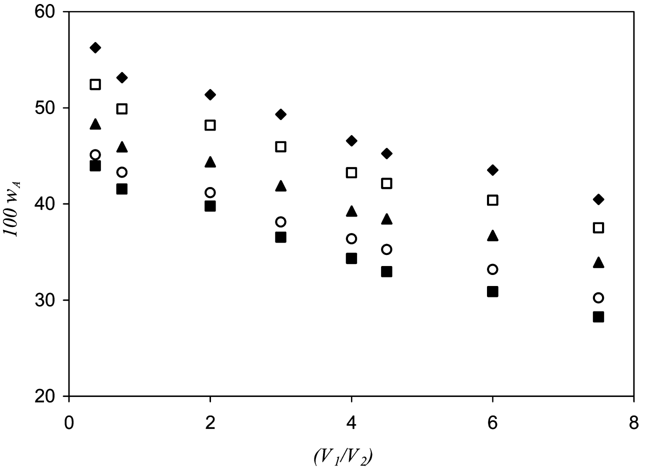

Figure 2. Effect of solvent-to-feed ratio on aromatic mass fraction, w_A, in extract phases at different temperatures: ◆, t = 50; □, t = 60; ▲, t = 70; ○, t = 80; ■, t = 90.

Table 3. Properties of Raffinate Phase for Furfural (1) + Lube-Oil Cut (2) System at Several Temperatures

		n	ρ (20 °C)		furfural		
t/°C	(V_1/V_2)	(20 °C)	g·cm^{-3}	$100w_S$	($100w_1$)	$100w_A$	$100w_N$
50	7.5	1.49092	0.8885	0.85	5.23	8.19	8.96
50	6	1.49366	0.8909	0.91	6.07	10.53	10.18
50	4.5	1.49738	0.8953	1.13	6.92	12.50	11.21
50	4	1.49881	0.8967	1.17	7.31	13.32	11.85
50	3	1.50218	0.9002	1.43	8.26	15.29	12.74
50	2	1.50623	0.9059	1.65	9.38	17.35	13.31
50	0.75	1.50922	0.9091	1.83	10.64	19.54	14.46
50	0.375	1.51543	0.9168	1.90	12.15	22.67	14.12
60	7.5	1.48859	0.8853	0.76	6.73	6.82	8.67
60	6	1.49177	0.8887	0.88	7.93	9.39	9.98
60	4.5	1.49565	0.8929	1.11	8.85	11.69	11.38
60	4	1.49682	0.8938	1.15	9.38	12.54	11.63
60	3	1.50123	0.8992	1.34	10.56	14.73	12.32
60	2	1.50498	0.9046	1.58	11.97	16.63	13.10
60	0.75	1.50807	0.9081	1.79	13.71	18.84	14.27
60	0.375	1.51345	0.9141	1.84	15.73	21.91	14.02
70	7.5	1.48687	0.8824	0.65	8.22	5.87	8.21
70	6	1.48975	0.8859	0.82	9.53	8.38	9.66
70	4.5	1.49318	0.8894	1.02	10.47	10.46	10.92
70	4	1.49446	0.8902	1.09	11.03	11.39	11.11
70	3	1.49841	0.8948	1.20	12.36	13.61	12.01
70	2	1.50185	0.8997	1.47	14.05	15.48	12.73
70	0.75	1.50513	0.9032	1.71	15.72	17.70	13.98
70	0.375	1.51009	0.9093	1.77	17.98	20.35	13.85
80	7.5	1.48513	0.8797	0.68	9.78	4.76	7.92
80	6	1.48812	0.8832	0.77	11.06	7.63	9.42
80	4.5	1.49183	0.8878	1.02	12.11	9.52	10.44
80	4	1.49378	0.8895	1.07	12.84	10.72	10.84
80	3	1.49751	0.8941	1.19	14.17	12.90	11.41
80	2	1.49966	0.8956	1.47	15.78	14.83	12.55
80	0.75	1.50293	0.8988	1.68	17.45	17.30	13.83
80	0.375	1.50868	0.9067	1.75	19.57	19.87	13.72
90	7.5	1.48352	0.8763	0.65	11.23	4.26	7.77
90	6	1.48619	0.8794	0.76	12.64	6.96	9.24
90	4.5	1.48896	0.8818	0.98	13.62	8.93	10.19
90	4	1.49078	0.8839	1.02	14.38	9.92	10.69
90	3	1.49482	0.8886	1.21	15.69	12.29	11.68
90	2	1.49703	0.8904	1.45	17.22	14.18	12.45
90	0.75	1.50034	0.8939	1.68	19.05	16.53	13.72
90	0.375	1.50461	0.8975	1.75	21.28	19.43	13.66

Regarding the reactivity of furfural with oxygen, furfural was distilled before utilization to remove the oxidation products formed due to contact with air. Furfural purity was 98 %, as was tested by gas chromatography (GC).

The experimental extraction setup consisted of a cylindrical stirred glass tank that was isolated from air by a Teflon bonnet. The system was equipped with a valve to remove the extract phase, a nitrogen cylinder, and a variable speed agitator (Figure 1). Temperature was regulated by a thermostatic bath (Shimifan S-57) and controlled within ± 0.1 °C.

In this experimental study, furfural and lube-oil cut were mixed together in several solvent-to-feed ratios at different temperatures. At the end of each run and formation of two immiscible phases, furfural was removed from the extract and raffinate phases by vacuum distillation. The agitation time in the experiments was 1 h at 430 rpm, and the settling time was 2 h. During the extraction experiments, a nitrogen stream was purged into the batch to prevent the oxidation of furfural.

The experimental values of viscosity and density are important parameters for lubricating oils. Also, by knowing viscosity values at (37.8 and 98.89) °C, the molecular weights of the samples could be calculated on the basis of ASTM D 2502-04.[24] This test method is a graphical procedure for determining molecular weight through the viscosity measurement.

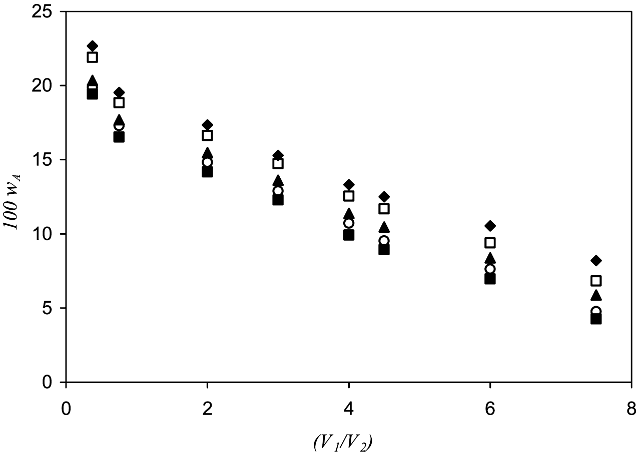

Figure 3. Effect of solvent-to-feed ratio on aromatic mass fraction, w_A, in raffinate phases at different temperatures: ◆, $t = 50$; □, $t = 60$; ▲, $t = 70$; ○, $t = 80$; ■, $t = 90$.

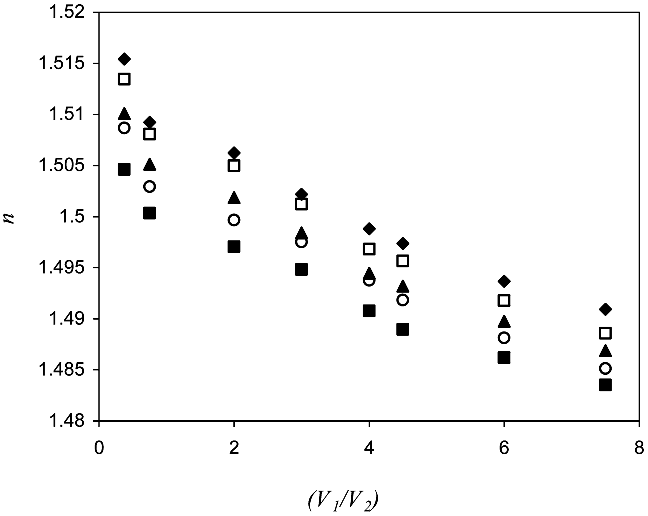

Figure 4. Effect of solvent-to-feed ratio on refractive index, n, in raffinate phases at different temperatures: ◆, $t = 50$; □, $t = 60$; ▲, $t = 70$; ○, $t = 80$; ■, $t = 90$.

The ASTM D3238[22] standard test method can be used for the characterization of petroleum mixtures. According to this procedure, by using density at 20 °C (d_{20}), refractive index at 20 °C (n_{20}), molecular weight, and sulfur mass fraction (w_S), the distribution of aromatic, naphthenic, and paraffinic compounds in hydrocarbon mixtures can be obtained. The above methods are described in the Appendix.

Viscosity and density were measured with an Anton Paar SVM3000 instrument according to ASTM D-7042. Refractive index (n) of samples was measured by an index instruments GPR-11-37E refractometer. The amount of sulfur in samples was determined with a Perkin-Elmer Optima-5300V analyzer. The uncertainty in measurement of viscosity was ± 0.01 pa·s; density ± 0.0001 g·cm^{-3}; sulfur mass fraction ± 1; and that of refractive index was ± 0.0001.

Results and Discussion

The experimental conditions and results of experiments are shown in Tables 2 and 3 for raffinate and extract phases. On the basis of the experimental values for raffinate and extract phases, the aromatic, naphthenic, and paraffinic content of each mixture was calculated by ASTM standards that were described in the previous section.

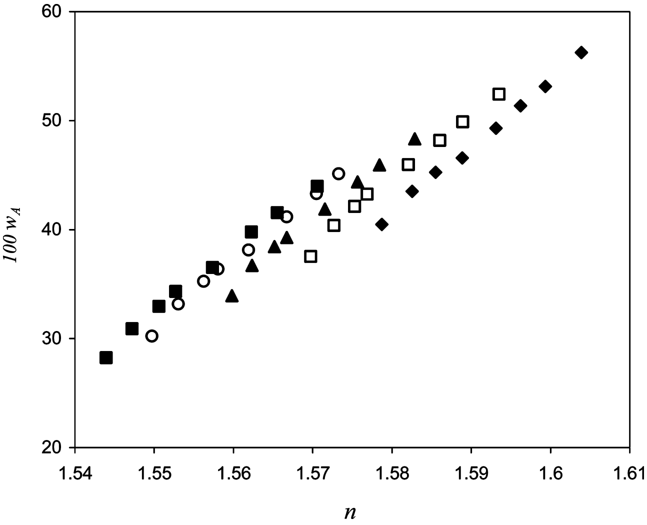

Figure 5. Effect of aromatic mass fraction on refractive index in raffinate phases at different temperatures: ◆, $t = 50$; □, $t = 60$; ▲, $t = 70$; ○, $t = 80$; ■, $t = 90$.

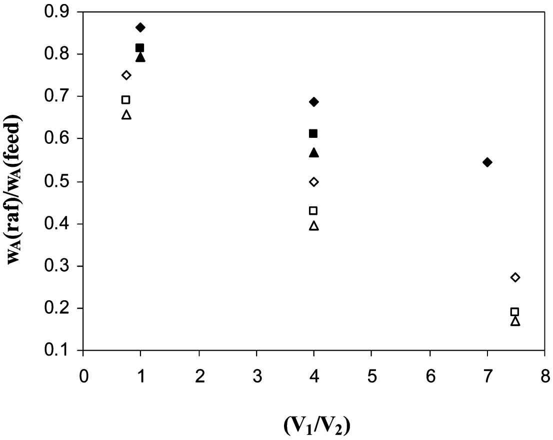

Figure 6. Effect of solvent-to-feed ratio on aromatic mass fraction in raffinate phases to aromatic mass fraction in feed, $w_{A(raf)}/w_{A(feed)}$: ◆, $t = 60$; ■, $t = 80$; ▲, $t = 100$ (Coto et al.[6] in 1, 4, and 7 solvent-to-feed ratios); ◇, $t = 60$; □, $t = 80$; △, $t = 90$ (this work in 0.75, 4, and 7.5 solvent-to-feed ratios).

Effect of Solvent-to-Feed Ratio. Table 2 shows w_A, w_N, and w_P for extract phases. Also, mass fraction of aromatics in extract phases is shown in Figure 2 for several solvent/lube ratios at different temperatures. Table 2 shows that by increasing the solvent-to-feed ratio, w_N is increased whereas w_A is reduced for extract phases, and w_P is virtually unchanged with increasing solvent/lube ratio.

Table 3 shows the values of w_A, w_N, and w_P for raffinate phases. In this table, w_A and w_N are reduced by increasing the solvent-to-feed ratio, whereas w_P is increased. This phenomenon could be related to the high tendency of furfural to dissolve the cyclic compounds. Therefore, by increasing the amount of solvent, the naphthenic and aromatic compounds began to dissolve more rapidly in solvent, and their amounts in the raffinate phase decrease. The aromatic content of the raffinate phase is shown in Figure 3 at several solvent/lube ratios and at different temperatures.

Tables 2 and 3 show that by increasing the solvent-to-feed ratio, the furfural contents of extract phases have an increasing trend for the extract phase and a decreasing trend for the raffinate phase.

Figure 4 shows the effect of solvent-to-feed ratio on refractive index for raffinate phases at different temperatures. The refractive index is directly related to the mass fraction of aromatic compounds, as shown in Figure 5 for the extract phase.

An attempt is made to compare the results of this work with previous data reported by Coto et al.[6] for light neutral distillate (LND). Because the feeds were different, a new parameter is defined as the aromatic mass fraction in raffinate to aromatic mass fraction in feed, $w_{A(raf)}/w_{A(feed)}$, to facilitate the comparison.

Figure 6 shows a comparison between the effect of solvent-to-feed ratio on $w_{A(raf)}/w_{A(feed).}$ Although the feeds are different, both lube-oil cuts show similar trends for variation of $w_{A(raf)}/w_{A(feed)}$ with solvent-to-feed ratio and temperature.

The results show that for LND, the ratios $w_{A(raf)}/w_{A(feed)}$ are about 25 % larger than those of this work. This deviation could be justified as follows: (1) The physical properties of feeds were different. (2) The aromatic content of LND was 48.5 %, whereas the aromatic content of our feed was 25.1 %. (3) LND and the accompanied raffinates had been characterized by ASTM D2007, but the feed and raffinates in this work are characterized by the more precise ASTM D3238.

Effect of Operating Temperature. Investigation of Tables 2 and 3 for extract and raffinate phases shows that by increasing the temperature, w_A and w_N are reduced and w_P is increased. Also, by increasing the temperature, the furfural content is reduced for extract phases and increased for raffinate phases.

Conclusions

The solvent-to-feed ratio and operating temperature are the main effective parameters on the extraction of aromatics and manufacturing of lubricating oils.

Increasing the amount of solvent (furfural) increases the yield of extraction of aromatic from lube-oil cut. Increasing the amount of furfural reduces the amount of w_N in lube oil and thereupon reduces the quality of product. By increasing the amount of furfural, the furfural content of the extract phase increases and imposes more costs for solvent recovery. The amount of aromatic content in the raffinate phase is reduced by increasing the temperature, but its furfural content increases at the same time.

Abbreviations

Nomenclature

M = molecular weight
n = refractive index
t = temperature, °C
ρ = density, g·cm^{-3}
w_i = mass fraction of component i

Subscripts

A = aromatics
N = naphtenics
P = paraffinics
S = sulfur

Appendix

On the basis of the measured values for n, ρ, and w_S, the values of aromatic, naphthenic, and paraffinic content were measured on the basis of ASTM D-3238,[22] which is expressed in the following paragraphs:

First, the factors V and W should be calculated from the observed ρ and the observed refractive index (n) by using the following equations

$$V = 2.51(n^{20} - 1.4750) - (\rho^{20} - 0.8510) \qquad (1)$$

$$W = (\rho^{20} - 0.8510) - 1.11(n^{20} - 1.4750) \qquad (2)$$

We calculated the percentage of aromatic hydrocarbons (w_A) from V and the molecular weight (M) by using one of the following equations

$$\text{If } V \text{ is positive:} \quad 100w_A = 430V + 3660/M \qquad (3)$$

$$\text{If } V \text{ is negative:} \quad 100w_A = 670V + 3660/M \qquad (4)$$

We can calculate the molecular weight of hydrocarbons by using ASTM D-2502.[24] This test method is a schematic method for determining the molecular weight by using the viscosity measurement.

In the next step, the percentage of ring structure (w_R) hydrocarbons (aromatic and naphthenic) should be calculated from w, sulfur content, and the molecular weight.

$$\text{If } W \text{ is positive:} \quad 100w_R = 820W - 300w_S + 10\,000/M \qquad (5)$$

$$\text{If } W \text{ is negative:} \quad 100w_R = 1440W - 300w_S + 10\,600/M \qquad (6)$$

For calculating w_N and w_P, the following equations were used

$$w_N = w_R - w_A \qquad (7)$$

$$w_P = 1 - w_R \qquad (8)$$

Acknowledgment

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